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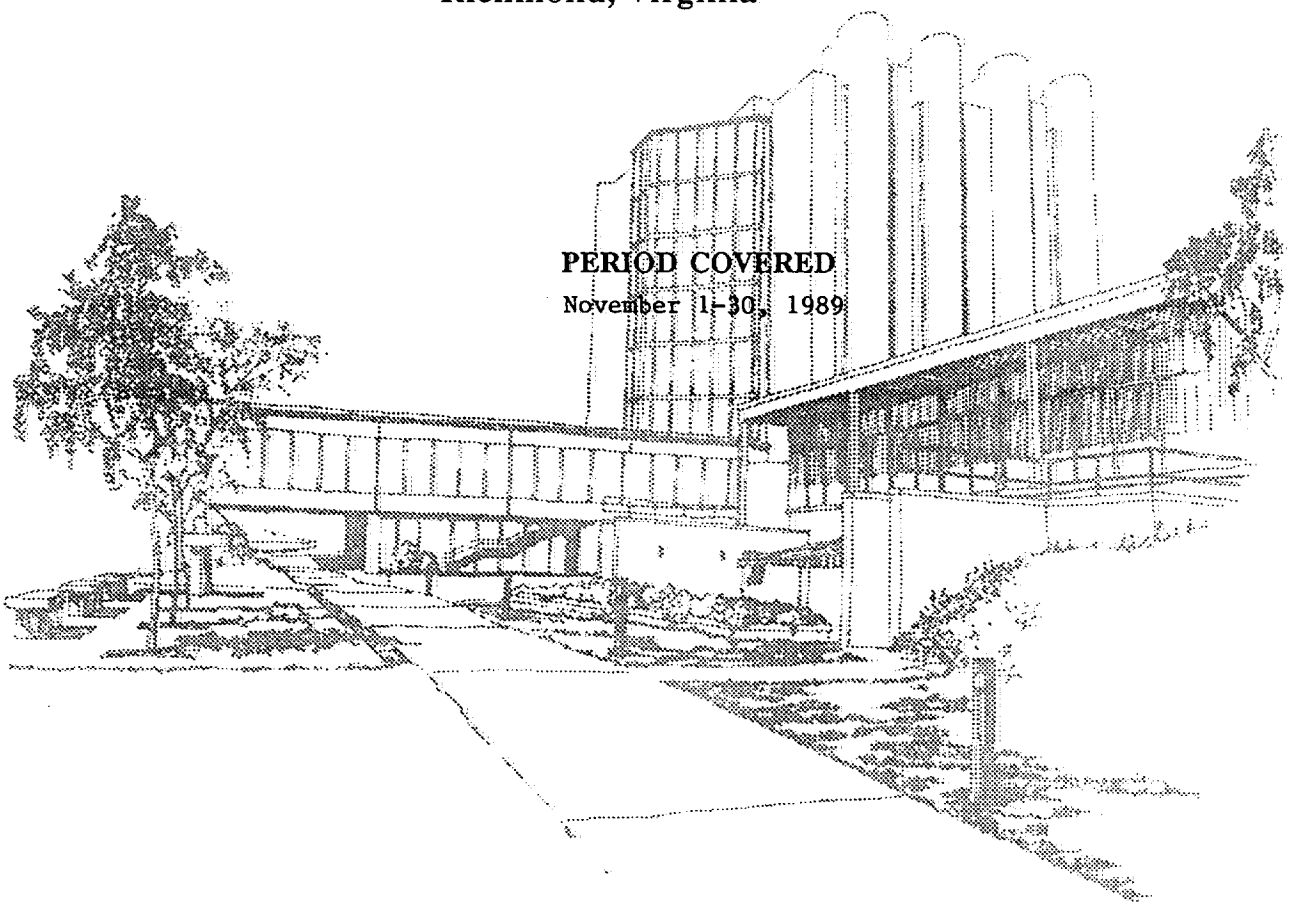

PERIOD COVERED
November 1-30, 1989

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*N/R = No Report

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PROJECT NUMBER: 2107
PROJECT TITLE: Filter Research & Development
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: November, 1989

I. IMPROVED FILTRATION - INCREASED EFFICIENCY:

A. Objective: Develop filter systems with a higher efficiency than presently available and evaluate them for subjective advantages.

B. Status: CA WEB: Analysis of the CA Web filter rods produced at Intertaba indicate that the rod to rod RTD coefficient of variation is between 10.6 and 12.3%. This is not as good as the numbers reported by Celanese (9%) for the same web material. Causes of this difference are being investigated.

NEW WEB TECHNOLOGY: Two samples of conventional paper which were experimentally acetylated and made into filter rods at Intertaba were subjectively evaluated by a small Flavor panel. The materials were described as not like CA or paper, but one of the samples was judged somewhat like CA. Equivalent delivery cigarettes with these filters are being prepared for further testing.

A sample of paper filter material and one of meltblown polypropylene web have been coated with a solution containing cellulose acetate and then dried. Filters will now be made from these materials for subjective evaluation.

Two versions of handsheets have been made from 75% CA/25% Soft Wood and 75% CA/24% Soft Wood/1% CMC (binder), spliced into rolls, corrugated and made into sample filters. These will now be subjectively evaluated in order to determine the effect of a combined CA/paper web.

Eastman has been asked to supply 300 lbs. of 2.0 dpf staple cut into 1/4" lengths for a pilot sheetmaking run at the University of Maine. Sheet properties of paper produced with 1/4" staple was comparable to those of paper made with shorter cut length.

PAPER FILTERS: Preliminary testing and modeling is being done in preparation for making a series of equivalent delivery cigarettes with CA, CA Web, and Paper filters for subjective characterization.

II. IMPROVED FILTRATION - MENTHOL STABILITY:

A. Objective: Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.

B. Status: As a result of discussions held under our secrecy agreement, Eastman has successfully spun filter tow with 3, 7, and 14% triacetin contained in the dope prior to spinning. This

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material has been made into filter rods by Eastman and will be fabricated into cigarettes by Semiworks in early December. The stability of menthol delivery in smoke will be examined as the cigarettes undergo ageing.

Analytical data on menthol delivery of filters made with 4% triacetin applied at 160°F is being received and will be evaluated for the possible stabilization of menthol delivery.

III. **IMPROVED FILTRATION - NOVEL FILTER SYSTEMS**:

A. **Objective**: Develop and evaluate new and unique cigarette filters which may offer a distinct product advantage.

B. **Status**: CONCENTRIC FILTERS: A dual cambridge pad holder has been fabricated which separates the inner and outer smoke flows from concentric filters and is being evaluated by CI personnel on a 20 port smoking machine. Initial results indicate a 80/20% outer/inner split for the filters tested. Additional trials are planned.

A series of seven concentric filter models have been ordered from Filtrona which cover a range of theoretical core to peripheral flow ratios. These will be evaluated analytically and subjectively to characterize their performance.

Smoke delivery of various filter systems has been videotaped and will be studied in an attempt to characterize the physical delivery of smoke to the subjective response.

RHODIA: Representatives of Rhodia's R&D Department presented an overview of their current programs to Philip Morris R&D. Their work with meltblown materials, additives, and their process of supercrimping are particularly interesting to us and will be pursued.

IV. **IMPROVED FILTRATION - SELECTIVE FILTRATION**:

A. **Objective**: Explore the use of specific additives in filters for selective filtration or subjective modification of smoke.

B. **Status**: CARBOWAX REPLACEMENT: PM Super Lights models have been made with a 30% more active carbon in an effort to reduce the weight of carbon per tip and still achieve the desired gas phase filtration. These are currently in CI for analytical testing.

ALTERNATIVE PLASTICIZERS: Models of Marlboro Lights cigarettes made in Semiworks with triethyl citrate and diethyl malonate plasticizers in the filter at 5 and 8% are currently being subjectively evaluated by the OC smoking panel. Additional plasticizers are being investigated.

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CARBON: A seminar was given by two researchers from Calgon Carbon describing the function of activated carbon as a filtration medium to a general audience on November 30 in the R&D lecture hall.

V. **FILTER SUPPORT FOR OTHER R&D PROGRAMS**:

A. **Objective**: Provide design assistance and potential new filter systems for other R&D programs.

B. **Status**: ART: Numerous ART models made with CA filters containing sodium carbonate, high denier tows, and paper filters are being evaluated by the ART Project team.

HIGH TASTE/LOW TAR: Two Project Lowvent cigarette models, targeted for 2mg tar at 35% ventilation, were made in Semiworks and are now in CI for testing.

LARK VENTILATION VARIABILITY STUDY: Analytical results of cigarettes made with a Kimberly-Clark porous combining wrap with a spiral hotmelt pattern and two Ecusta porous wraps with a patterned hotmelt show a significant decrease in ventilation variability compared to production Lark Milds KS cigarettes. The best Ecusta wrap achieved an average ventilation of 40% (control 24%) and a coefficient of variation of 14% (control 25%). Models produced with the ventilation into the cavity were better, with a ventilation of 49% and a COV of 8%. The machinability and hot collapse performance of this wrap is equivalent to the current material. This Ecusta wrap has been recommended as a replacement to the current mechanically perforated wrap which is now yielding highly variable product.

Operations Services and the Japan Product Development Group will conduct an additional trial prior to implementation. Development of the Kimberly-Clark wrap will be pursued as a second supplier.

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PROJECT NUMBER: 2108
PROJECT TITLE: New Product Technology
PROJECT LEADER: W. T. Callaham
PERIOD COVERED: November, 1989

I. SAUNA

A. **Objective**: Develop products utilizing a novel filter system.

B. **Status**: As a result of last month's meeting with EEMA personnel to discuss product modifications for better competing against Barclay, several new models were made and tested with a tar target of 4mg. These models all used a new Sauna blend having lower available tars and the cigarette length was changed from 79mm to 84mm. The filter systems selected were a conventional Sauna-type, a dual plastic fluted filter with a CA or concentric segment, and a straight concentric filter. The cigarettes were smoked by the ISO DIN method and with the new Coresta holder. All models delivered more than the target 4mg tar. Thus far, eight different dual models were subjectively screened and two have been selected for further evaluation.

Evaluation of these models using the Coresta recommended cigarette holder indicated higher variability compared to the DIN average delivery than for Barclay. Additional smokings with the holder are warranted to better understand this. In support of this work, more holders have been requested from FTR.

Arrangements have been made to have one of the Semiworks combiners reconfigured for an 18mm/9mm filter during a coincident refurbishment at a local machine shop. This combination is necessary for a dual fluted/charcoal filter for a Japanese Sauna (target 5mg tar). Additional fluted filters with the proper circumference have been made in Louisville to support this.

Fabrication by the R&D machine shop is almost complete on a bench-top machine to produce blocked fluted filters. Debugging operations are currently in progress.

Astra filters were ordered from Filtrona for evaluation as a substitute for the plastic fluted filter. These filters have a corrugated paper perimeter.

B. **Plans**: Finalize construction of the most subjectively acceptable 4mg model and submit for EEMA approval.

Manufacture a working Japanese Sauna model with various dual filter systems using Sauna, plastic fluted or concentric filters.

Evaluate the Filtrona Astra filter as a substitute for the plastic fluted filter.

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II. MACHINERY DEVELOPMENT

A. **Objective**: Develop or modify equipment to facilitate the development of new products.

B. **Status**:

Paper Coating: Two new gravure rollers for printing sol-gel on paper were fabricated. The rollers have patterns with alternating bands of either 3, 4, or 5mm width and 6, 7, or 8mm width, both with a 21mm repeat.

A trip was made to Western Michigan University to review their printing and coating capabilities and to assess various equipment for potential installation at Philip Morris. A decision was made that our equipment in-house is adequate for our needs, however, some modifications may enable higher coating levels.

Embossing: An improved "pocket" foil, produced at Intaroto, was evaluated by Project Sigma personnel and found to be too low in porosity. Consequently, another tool was fabricated to double the open area of the foil and samples are currently being produced for further evaluation.

Tubewinding: The tubewinder and portable foaming machine have been overhauled and are currently undergoing startup debugging operations for Project Sigma.

Web filter making: Mechanical improvements were made to the web filter machine to better facilitate running paper or CA web with porous plug wrap. These include the addition of a PZ spray booth, a speed controller on the corrugator, and a hot melt application system. Startup assistance was provided by Intertaba personnel during a coincident visit to PM.

C. **Plans**: Pursue modifications to our existing coating and printing equipment to facilitate higher additives levels on papers.

Evaluate new "pocket" foil samples.

Complete startup of the tubewinder/foaming unit and produce samples as requested by Project Sigma.

Continue development of web filter maker improvements to facilitate better quality web-type filters.

III. HUMIDOR PACK

A. **Objective**: To develop a moisture release device for use in a cigarette pack which maintains pack OV at a desired level.

B. **Status**: Humidor packets were produced at Paco Pharmaceuticals for a Middle East shipping simulation study to be conducted in house.

C. **Plans**: Conduct the shipping simulation study.

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IV. ASH TRAY ODOR

A. **Objective**: Develop products which facilitate the elimination of ash tray odor.

B. **Status**: A quote was received from the Dabney Corporation for mass production of a plastic odor-less ash tray. Up-front capital and four months is required to produce the molds. Another quote is being sought. This information was transmitted to New York and at the same time, they requested a slightly downsized, redesigned version. A new prototype is being designed. Currently, we have chosen not to pursue a patent.

Virginia Slims Lights cigarettes were injected with hexyl cinnamic aldehyde into the filters for odor panel testing of the spent filters.

C. **Plans**: Complete fabrication of the new ashtray and forward to New York for comments.

Complete odor panel testing of the injected filters and develop a working filter system to accomplish the same end result.

V. MENTHOLATION TECHNOLOGY

A. **Objective**: To develop alternative mentholation techniques which offer a product or processing advantage.

B. **Status**: A second study of mentholated inserts was initiated to analytically and subjectively evaluated cigarettes with higher menthol levels on the paperboards and combinations of mentholated inserts with MOF. The test models include Marlboro Lights 100 SP where one has all of the required menthol on two inserts and the other has the required menthol split between one insert and the foil. Samples are currently being aged.

A meeting was held with Packaging Engineering personnel to discuss the feasibility of this approach. Several design schemes were assumed and cost/time estimates were made. Additional studies are being planned to investigate mentholation of the box innerframe and placement of inserts outside of the foil.

Training was completed for Semiworks personnel on operation of the PM mentholation unit and operational documentation was turned over to them.

C. **Plans**: Complete the mentholated insert study.

Investigate new embossing patterns for high basis weight foils so that higher menthol loadings may be applied to the foil.

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VI. FILTER R&D SUPPORT:

A. **Objective**: To support the Filter R&D group in providing improved filter systems which offer product advantages.

B. **Status**:

Concentric Filters: A cambridge pad holder for concentric filter cigarettes was fabricated and tested. This holder allows for the collection of TPM from both the core and periphery of the filter. Minor design modifications are required to optimize its performance.

FML: Assistance was provided to FML at Bridon, England in helping them resolve some operational problems such as weight control and recessed ends.

CA Filter Additives: Citric acid analyses of CA filter rods prepared using the Mark-6 filter flavor addition system indicated slightly lower levels of citric acid per plug than targeted. Adjustments will be made and a capability trial will be conducted again.

Paper Filter Study: Samples of paper filters were produced with levulonic acid, mono-ammonium phosphate, and potassium carbonate as part of a program to develop a subjectively acceptable paper filter.

C. **Plans**: Complete optimization of the cambridge filter pad holder for concentric filters and pursue routine use.

Optimize performance of the liquid additive system on the Mark-6.

Produce web-type filters as requested by Filter R&D personnel.

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PROJECT NUMBER: 2305
PROJECT TITLE: Applied Flavor Investigation
PROJECT LEADER: J. W. Swain
PERIOD COVERED: November, 1989

I. PROJECT ART:

ART-By-Product Utilization:

A. **Objective**: To evaluate and develop process modifications for the utilization of ART process by-product tobaccos.

B. **Status**: Flavor modification tests of direct ART stem utilization (ES or IS) have been suspended due to change to lower CPI on stem. Tests are continuing to determine an acceptable level of substitution in sheet materials.

Earlier tests of large-scale Semiworks trials of blended RCB's which simulated low levels of MPC stem inclusion showed analytical differences. Since these variations may have contributed to subjective differences, final confirmation of acceptability of 2-3% MPC stem in RCB is in progress on a test production RCB. Subjective evaluations of 100% and 24% RCB handmade cigarettes look promising for the low level of MPC stem. Similar tests are in progress to also utilize Bermuda Hundred stem with citric acid. RCB incorporating 2% MPC and 2% citric acid BH stem is under evaluation which would utilize the current inventory within 13 months.

C. **Plans**:

Complete Panel Tests of RCB with BH Stems	December, 1989
Evaluate Lower Levels of BH Stem in Pilot RL's	December, 1989

II. OPERATIONS SUPPORT:

PROJECT GRAIN:

A. **Objective**: To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

B. **Status**: Cambridge cigarettes with control versus test AC (30% reduction in alcohol) from a Cabarrus production trial showed no differences on MC panel. However, initial panel comparisons had failed to show parity and further tests are planned with AC to be prepared at the Flavor Center.

C. **Plans**:

Install Sonolator System at Flavor Center	December, 1989

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Test AC's through Semiworks
Trials — January, 1990

III. **DRY FLAVOR REPLACEMENT**:

A. **Objective**: To develop, evaluate and establish specifications for dry flavor replacements.

B. **Status**: The Park 500 production trials with Dry Flavor replacement in RLB have been completed. Control and test RLB's were comparable on Park 500 analyses and initial subjective evaluation of 100% RLB cigarettes. Marlboro cigarettes have been requested to compare control and test RLB's at normal and 24% levels on internal panels.

C. **Plans**:

Complete Internal Panel
Evaluations — January, 1990

IV. **CASING TECHNOLOGY**:

A. **Objective**: Develop and evaluate casing systems and processing conditions to maintain and/or enhance subjectives.

B. **Status**: Evaluations continue on PG/G Free trials to determine physical and subjective differences. Complete analytical data anticipated in December.

Further casing evaluations on DL, 215 and 220 blends from Bermuda Hundred included tests of botanicals, Cooked Flavors, salts and compounded commercial flavors in efforts to increase response. Several casing trials were completed at Bermuda Hundred on different blends for POL tests.

C. **Plans**:

Summarize PG/G Free
Testing — December, 1989

Continue Casing Development
for BH Trials — December, 1989

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PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization/International Support
PROJECT LEADER: W. R. Bell
PERIOD COVERED: November, 1989

I. MARLBORO STANDARDIZATION:

A. **Objective**: Analytical and subjective evaluations of production Marlboro KS/LS.

B. **Status**: Test results for POL tests 3622 through 3626 have been received. All analytical data has been completed and presented to Manufacturing Services. Marlboro Lights pickup 11-8-89 is currently being analytically and subjectively evaluated.

C. **Plans**: Continue subjective evaluation on Marlboro Lights cigarettes.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL:

A. **Objective**: To provide subjective direction for programs with R&D and manufacturing locations.

B. **Status**: Eight (8) panels were completed for the reporting period. Panel has been evaluating ART prototypes for the month of November.

C. **Plans**: Provide support as needed.

III. INTERNATIONAL SUPPORT:

A. **Objective**: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

B. **Status**: Twenty-seven (27) panels completed during the reporting period.

PAN ASIAN MENTHOL - PROJECT CEDAR

A. **Objective**: Development of free-standing menthol model to compete with Salem Lights (Hong Kong).

B. **Status**: The second Cedar test vs. Salem Lights was overtipped and shipped.

C. **Plans**: Awaiting PMI test results (early December).

MERIT LIGHTS MENTHOL (JAPAN):

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A. **Objective**: Develop a Merit Lights Menthol for the Japanese market.

B. **Status**: The three test models and the Salem Lights (Japan) were overtipped, subjectively evaluated and released for shipment.

C. **Plans**: Awaiting Danchi test results.

CHESTERFIELD (JAPAN):

A. **Objective**: Develop a Chesterfield prototype for the Japanese market to compete with Lucky Strike.

B. **Status**: Promising results received from Danchi Panel testing. Flavor modification are in progress for additional Danchi and PMI testing in January, 1990.

C. **Plans**: Flavor modifications are currently under investigation.

DERBY MENTHOL (GUATEMALA):

A. **Objective**: Modification of Derby (Menthol) to compete with Casino (BAT) in the Guatemalan market.

B. **Status**: A revised "Casino-type" flavor was developed and shipped to Guatemala.

C. **Plans**: Subjective evaluations of the revised flavor will be conducted in Guatemala, as well as Richmond.

PROJECT BOLIVIA (EL SALVADOR):

A. **Objective**: Develop a cigarette to compete with "Delta" (BAT) brand in El Salvador.

B. **Status**: An aftercut flavor was developed to compete with BAT's Delta and was shipped to Guatemala for evaluation.

C. **Plans**: Evaluate prototypes when available, and make any necessary flavor revisions.

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PROJECT NUMBER: 2307
PROJECT TITLE: Basic Flavor Investigation
PROJECT LEADER: R. W. Hale
WRITTEN BY: B. A. Demian
PERIOD COVERED: November, 1989

I. BASIC FLAVOR INVESTIGATION:

A. Objective:

To develop basic and applied knowledge for the purpose of flavor improvement or modification of existing products and to provide the basis for development and application of flavor technology for new, unique products.

B. Status:

1. ART:

Several impact enhancers evaluated, with some success, including: botanicals, tobacco extract fractions and flavor chemicals.

Pyrolysis/HRGC/sniffing evaluation of Size Exclusion Chromatography Fractions of Tobacco Extracts: Peak I, II and III of E33 Bright Extract, Peak I and II of C34 Burley Extract, completed. The system consists of: Pyroprobe 120 pyrolysis unit, HP 5880 GC, CP Sil 5CB 10m*0.53mm ID column, open split 1:1 to FID detector and sniffing port.

Summary of findings:
- Peak I and II, regardless of provenience, are very similar in HRGC and odor profiles.
- Pyrolysis temperature: 650°C or 900°C yields practically identical odor profiles and very similar HRGC profiles.
- Peak I yields at pyrolysis a significant number of mercaptan-like peaks and a sizable number of smoky-phenolic peaks.
- Peak II releases a whole gamut of pyrazinic peaks and many smoky-phenolic peaks and has the highest intensity relative to sample size.
- Peak III, the weakest overall, yields mainly pyrazinic peaks.
- Peak II, Bright or Burley, appears to be the most promising for use as flavor enhancer.

This work has relevance for other projects, like Low Tar/High Flavor and others.

2. Low Tar/High Flavor:

The subjective evaluation of model cigarettes with various ventilation levels is completed. A summary of findings is being prepared.

II. ANALYTICAL SUPPORT:

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A. **Objective**: To provide analytical support for activities related to development and application of flavoring materials.

B. **Status**:

1. **External Analytical Support**:

Six samples of filler analyzed for anethole content. Training of a technician provided for anethole in filler analysis, in expectation for CTS to provide this analysis in near future.

2. **Cartier**:

Volatiles in box end caps determined for supplier qualification.

3. **Analytical Support**:

GC profiles of flavor 75-644.
GC profile of "Brasway" menthol, compares favorably to Brazilian menthol.
Headspace GC/MS and GC/IR of four samples of extruded tobacco.
Headspace/GC for residual solvents in 2 samples of St. John's Bread extracts.
Casings (3), flavor concentrates (2) and A/C flavor (1) analyzed by GC and HPLC for major components.

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PROJECT NUMBER: 4009
PROJECT TITLE: Development Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: November, 1989

I. REDUCED SIDESTREAM CIGARETTES:

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. Status:

Trim V: Three new inner liners from Papeteries de Mauduit were evaluated in 19.5 mm circumference models. The new papers were all less porous than the inner liner used on Superslims, and one contained 4% mono-potassium phosphate. They all achieved comparable visibility reductions at about 75%. Another set of 19.5 mm circumference cigarettes were smoked subjectively. The control double-wrap model was compared to one with phosphate on the outer wrap and one with phosphate on the inner liner. All models were considered different but acceptable, and they all gave visibility reductions of 75-78%.

Lotus: A meeting was held with representatives from Kimberly-Clark to discuss optimizing the heavy basis weight paper currently in use for development of Lotus. A matrix of papers was subsequently provided that consisted of samples containing 26 and 36% $CaCO_3$, each at 2.9 and 4.2 Coresta. Chemical Research provided target levels of KH_2PO_4 for each paper to achieve the desired visibility reductions for each paper. The papers were sized with the appropriate levels of additive for cigarette production.

Cigarettes made with varying ratios of potassium and phosphorous on the high basis weight paper showed increasing visibility reduction as the level of phosphorous increased, primarily due to a decrease in burn rate. A model with a low level of phosphoric acid was not subjectively different from the control containing 10% potassium phosphate. The control model, at tar delivery level of 8 mg, was put on the MC panel for subjective evaluation.

Cigarettes were made with three different $Mg(OH)_2$ papers from Ecusta, each with different burn additives, and each perforated and unperforated. The sidestream visibility reduction ranged from about 65% to about 75%, but the ash quality was typically poor for $Mg(OH)_2$ papers.

Ambrosia: Work has continued on including Aromatek-245 on the 73 g/M paper. Cigarettes with Aromatek-245 have been made in 24.8 mm circumference with the K-C heavy paper, in a double-wrap configuration, and using succinate as the fluxing agent; models were made with Marlboro filler and the new low sidestream blend. Several models have been evaluated subjectively. Sidestream

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visibility was reduced by 65% for all of the models except one with a phosphate double-wrap, which gave higher reduction.

C. **Plans**: Continue evaluating the effectiveness of different fluxing agents at varying levels.

Continue evaluating cigarettes with Aromatek-245.

Initiate development of a Marlboro-type cigarette with 40-50% visibility reduction.

Evaluate a new aftercut provided by Flavor Development for Lotus.

II. SIDESTREAM VISIBILITY MEASUREMENTS

A. **Objective**: Determine sidestream visibility of experimental cigarettes and commercial brands.

B. **Status**:

Sidestream Visibility Measurements: An experimental cigarette made with an overall calendered paper was compared to the control uncalendered 15 Coresta paper. There was a 30% reduction due to the calendering process. Static burn time increased by 20%.

C. **Plans**: Continue to test experimental low sidestream models.

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PROJECT NUMBER: 2304, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: G. N. Yatrakis, J. B. Easley and J. L. Spruill
PERIOD COVERED: November, 1989

I. LOW TAR/HIGH FLAVOR:

A. Project 605

1. **Objective**: Develop a 6 mg free-standing cigarette which appeals to flavor low smokers.

2. **Status**: Requests for prototypes are being submitted using Blend 218 (Project 202) with several filtration systems.

3. **Plans**: Analytical and subjective testing, evaluation of new filters as available.

B. Project 202

1. **Objective**: Develop a 2 mg product subjectively comparable to Merit Ultra Lights.

2. **Status**: The Blend 3 low ventilation models were completed and are in analytical. Filters used were paper/1.6 dpf CA and CA-web/1.6 dpf CA. Blend 218 models, with or without aftercut, were subjectively preferred over Blend 3.

3. **Plans**: Additional pre-expansion casing, blend casing, aftercut development work and filter evaluation.

C. Novent

1. **Objective**: Develop a 6 mg tar cigarette without filter ventilation.

2. **Status**: Blend A models using Carbon on paper/CA and Carbon on paper/CA-web filters have been made. Subjective evaluation is in progress.

3. **Plans**: Complete analytical and subjective evaluations, continue aftercut development and evaluation of additional filtration systems.

II. LOW SIDESTREAM:

A. Trim Two:

1. **Objective**: Development of 19.5 mm circumference products with 70% sidestream visibility reduction with and without sidestream aroma modifications.

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2. **Status**: Samples have been made using three blends. Additional samples were made on the Pilot Maker to evaluate coated outer wrap and alternative inner wraps using two blends.

3. **Plans**: Complete analytical and subjective evaluation. Issue preliminary specifications.

III. PROJECT AMBROSIA:

A. **Objective**: Develop a 23.0 mm circumference aromatic sidestream product and apply this technology to other products.

B. **Status**: POL 0511 was completed with results indicating that the Ambrosia model (100 mm, 24.8 circumference using Aromatek-245 via paper coating) was perceived as less intense.

A series of models using Aromatek-245 (paper coating) and -150 (via sideseam adhesive) with aftercuts that provide a mainstream sweet taste have been made. These models are being subjectively evaluated.

A new prototype using CR-2852 (vanillin release) coated on paper, in a Virginia Slims Lights configuration, was made. This model has shown subjective promise. A 100 gm sample of CR-2852 was requested by mid-December for prototype evaluation as well as Phase III clearance of the compound.

National Starch has been supplied with 4.5 lb. of Aromatek -150 to produce a small-scale production batch (10 gallons) of adhesive with 5% loading of -150. This material will be used to evaluate the application rate. Models will be produced with varying flow rates and analyzed for the quantity of -150 per cigarette. The models will be subjectively evaluated to determine the target application rate. National Starch has also been asked to study adhesive separation and viscosity stability at various conditions for this adhesive.

Requests for model making are being submitted using low-sidestream paper (73 gm basis weight) in conjunction with Aromatek -150 and -245. Models will be made at 24.8 mm circumference, 85 and 100 mm total length, with tar targeted between 10-11 mg. The -245 will be used at a 2% solution concentration level applied to paper and -150 will be applied at a 5% level in sideseam adhesive.

IV. PROJECT MARLBORO:

A. **Objective**: Design and develop Marlboro products with reduced tar (15 mg) and test 80 mm FTB vs. 83 mm FTB in 16 mg tar levels.

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B. **Status**: The first series of POL's for 80 mm and 83 mm @16 mg tar have been produced and shipped to panelists. They were POL #0320, 0321, 0323 and 3632. Two POL's, #0236 and #0232, for the non-menthol monadic panel will be produced in late December for shipment in early January. The 15 mg tar vs. 16 mg tar for 80 mm and 84 mm will be produced in early January and shipped when panelists are available.

C. **Plans**: POL test 15 mg models of 80 mm, 83 mm and 84 mm starting in January, 1990.

V. **PROJECT BASIC (BRISTOL)**:

A. **Objective**: Develop a generic product at the lowest possible cost to compete with Pyramid.

B. **Status**: Bristol production began in Louisville on November 27 for KS and 100's Lights Regular. Menthol versions began production November 29, 1989. Subjective evaluations appear to be acceptable. No analytical results are available at the present time.

C. **Plans**: Continue to monitor factory start-up of production.

VI. **PROJECT TARGET (BUCKS)**:

A. **Objective**: Develop Full Flavor and Lights Camel-like value entry candidates.

B. **Status**: Target II POL #3627 using Famous 2A blend in Full Flavor configuration was produced and shipped to panelists the week of November 2, 1989 to be tested against Camel KS. The Lights POL's (#4461, 4462 and 4463) were cancelled.

Target III using new blend #214 was designed and developed for the factory production start-up. Bucks Full Flavor and Lights began production at Stockton Street on November 14, 1989. Subjective and analytical evaluation show that the product specifications have been met.

C. **Plans**: POL #3633 has been produced to test Target III vs. Camel KS and will be ready to ship December 1, 1989. Monitoring of factory production runs will continue.

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PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PERIOD COVERED: November, 1989
WRITTEN BY: C. B. Altizer & R. E. Tinker

I. BATTISTONI (Asia Duty Free)

A. **Objective**: To develop an 83mm FTB product for the Asian duty free market.

B. **Status**: Preliminary specifications have been written.

C. **Plans**: A factory trial is being scheduled in December pending receipt of packaging materials and tipping paper.

II. L&M MILDS (Taiwan)

A. **Objective**: Develop a L&M Milds product for the Taiwan market.

B. **Status**: A factory trial has been conducted and specifications have been written.

C. **Plans**: A 90 million cigarettes export order has been placed with production scheduled December 3rd at the Manufacturing Center.

III. MARLBORO LIGHTS MENTHOL (Hong Kong)

A. **Objective**: Increase the delivery of the smoke menthol from 0.20mg to 0.35mg in January, 1990, and to the standard 0.50mg at a later date.

B. **Status**: A factory trial is being scheduled to determine the correct level of mentholated foil needed to achieve the new smoke menthol target.

C. **Plans**: Complete analytical testing and revise specifications for pending orders.

IV. PROJECT RING (Korea)

A. **Objectives**: Develop a family of products (approximately 4mg, 6.5mg, and 9mg tar) acceptable to mainstream Korean smokers.

B. **Status**: At the November CPC meeting, the 9mg Marlboro Lights KS and 100mm versions were approved for export. Specifications were written.

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C. **Plans**: Evaluation of additional Ring prototypes will continue on the Seoul Consumer Panel format. Two additional consumer tests have been requested. Test #4 will include Marlboro Lights, an Ultra Lights prototype @ 6mg, and two Ring prototypes @ 6mg and 4mg. Test #5 will test 88 Lights and three Ring prototypes @ 8.5mg, 6mg, and 4mg.

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PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PROJECT LEADER: S. B. Nelson
PERIOD COVERED: November, 1989

I. LARK 1989 PROGRAM

A. Objective: To optimize the subjective acceptance of the Lark family while retaining the Lark character.

B. Status: The results of the Lark family blend/tar evaluations were reviewed with PM Asia management. The test results indicated the Mount Blend and Lucky Strike were equally preferred and rated by Mild Seven and Mild Seven Lights smokers. These smokers preferred and rated higher Lucky Strike over the current Lark blend. A test program was developed to evaluate a milder American-type blend -- Starship -- with a P-S-P filter. Results are expected in March, 1990.

Based on Danchi test results it was decided to lower the FTC tar of Lark family products as follows:

Lark	15.5 to 14.5 mg/cigt.
Lark Milds	11.5 to 10.5 mg/cigt.
Lark Super Lights	8.0 to 7.5 mg/cigt.

These tar reductions will be initiated in January, 1990.

II. CHESTERFIELD

A. Objective: To develop an 11 to 12 mg tar American blended product.

B. Status: The Danchi results of the Chesterfield model with Mount blend, L&M Milds and Lucky Strike were received and analyzed. The liking ratings for Lucky Strike and L&M Milds were unusually low and this test will be repeated. However, the liking ratings do indicate the Chesterfield model should be pursued further.

In addition to the repeat of the Danchi test, three PMI tests have been scheduled.

- L&M Milds versus Lucky Strike
- Chesterfield Model versus Lucky Strike
- L&M Milds versus Chesterfield Model

These tests will be conducted among Mild Seven, Mild Seven Lights, and Seven Star smokers. Results of the repeat Danchi and the PMI tests are expected in March, 1990.

III. JAPAN MARLBORO

A. Objective: Maintain and/or enhance the subjective analytical and physical performance of Japan Marlboro.

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B. **Status**: Top-line PMI test results of Japan Marlboro @ 16.4mg FTC tar and Lucky Strike @ 11.5mg FTC tar were received. This test, which was conducted among cross section smokers, showed a preference and higher rating for Lucky Strike. However, significant improvement was noted between this test with the new Japan Marlboro (charcoal filter) and a previous test of old Japan Marlboro (no charcoal filter) versus Lucky Strike.

The following Danchi tests have been scheduled with results anticipated in March, 1990:

Test 1

- Marlboro KS SP
- Marlboro KS Box
- Lucky Strike KS SP
- Lucky Strike KS Box

Test 2

- Marlboro Lights KS SP
- Marlboro Lights KS Box
- Camel Lights KS Box
- Kent Milds KS Box

In discussions with JTI it was agreed that they would utilize their existing filter inventory and convert from carbowax to their triacetin in January, 1990.

It was also agreed that they would order skip-gap glue rollers immediately for the introduction of ventilation on Marlboro KS and 100's. It is now planned to introduce ventilation on Marlboro in late February, 1990.

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PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: November, 1989

PACKAGING STUDIES

A. **Objective**: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Status/Plans**:

Residual Printing Solvents in Packaging Materials: Coordinated evaluation of the following items:

- Merit KS FT 14/210 (1D-069A)
- J&B Decor Green Ink AOBL-36872
- BASF Aquagem Champagne Gold 89S559
- Alcohol Resistant Foil Innerliner Vehicle (GB)
- B&H DUL 20/200 (2C-925A/RG)
- B&H DUL Men 20/200 (2C-935A/RG)
- Marl 100 Gold SP 20 (4L-981A/JWF)
- Marl LS FT 20/200 w/Water Lacq. (50-904A/VFB-Tn)
- B&H 100 SP 20 Roll Emboss (2L-439A/IPC)
- Bristol Lts Reg 100 SP 20 (26-872A/JWF)
- Bristol Lts Men KS SP 20 (26-871A/JWF)
- Bristol Lts Reg KS SP 20 (2L-870A/JWF)
- Bristol Lts Men 100 SP 20 (2L-873A/JWF)
- Bristol Lts Men 100 SP 20/200 (2C-868A/RG)
- Bristol Lts Reg 100 SP 20/200 (2C-867A/RG)
- Bristol Lts Reg KS SP 20/200 (2C-869A/RG)
- Bristol Lts Men KS SP 20/200 (2C-870A/RG)

6's Carton Overwrap: A six week storage study is underway, in conjunction with Quality Engineering, to determine the impact of eliminating carton overwrap on 6's.

Cartier: Supply of Phillips' 55500 PE resin, for 10's end caps, has been jeopardized due to an explosion suffered at one of their facilities. The following four alternates are being evaluated for acceptability:

- Quantum LA3550B PT
- Soltex Fortiflex T50-4400-119 PE
- Amoco #10-7844 PP
- Himont Pro-fax PD701 PP

Foil with Alkaline Process Paper: Marlboro Lts KS SP cigarettes were packaged at the semi-works with test and control foil bobbins processed on the PM mentholator. Samples are currently undergoing two weeks menthol equilibration prior to storage, spotting and staining, analytical, and subjective testing.

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Source: https://www.industrydocuments.ucsf.edu/docs/zmjm0000

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: November 1989

I. Molten Menthol Application to Foil

A. Objective: Evaluate the use of a molten menthol applicator as a means of applying menthol to foil without the ethanol carrier.

B. Results: A Job Order Request has been submitted for the acquisition of a molten menthol application system capable of operating at production speeds. The system is based on a Grayco LTI hot melt adhesive applicator system.

A test is in progress to determine how much menthol will transfer to the face of a strip of uncoated foil placed in a bed of coated foil. There was no visual evidence of menthol transfer to the uncoated strip when the foil was stored at ambient conditions. However, when the foil was stored in the desert room, there appeared to be as much menthol on the face of the uncoated strip as there was on the coated foil. Samples have been submitted for analytical testing to verify these observations.

C. Plans: Compare the relative buildup of menthol on rollers in contact with MOF and MMOF. Purchase a hot melt adhesive applicator capable of accurately metering molten menthol onto foil at production rates. Establish the operating conditions which minimize the retention of menthol on the foil face. Determine if MMOF reduces the formation of esters in packed cigarettes compared to MOF.

II. MGC Application

A. Objective: Assist New Products in producing cigarette samples containing the MGC menthol release compound by investigating the feasibility of applying MGC formulations to filler in a maker chimney using a foam bound rod-type nozzle and a high pressure pump.

B. Results: The initial MGC production run at Lee Laboratories was contaminated during processing and had to be discarded. MGC should be available from a second production run by the first week of December.

Installation of an agitated thin-film evaporator for use in MGC concentration trials is in progress. Assembly of the off-line pumping/metering system has been completed.

C. Plans: Develop MGC formulations suitable for applying in a maker chimney. Check out the operation of the off-line pumping/metering system to be used for preliminary tests.

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PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: November, 1989

I. ART STEM UTILIZATION

A. **Objective**: Develop the capability to fully utilize spent absorber stems from the ART process.

B. **Results**: Blended cigarettes containing reduced levels of RCB made with 11% monopotassium citrate (MPC) stems were screened by the Richmond Panel. The model with test sheet replacing 50% of the RCB (simulates 5½% MPC stem in RCB) gave differences (heavy and mouthcoating) vs control; the bitter/green aftertaste associated with higher ART stem levels was not detected. The model replacing 25% of the RCB (simulates ~3% MPC stem in RCB) gave only minor differences (not typical ART stem notes); picking data showed 28% recon in the test cigarette vs 20% in the control, which would explain the lower (Δ=1 mg) tar delivery in the test cigarette and would account for the subjective differences noted. The panel recommended making a legitimate RCB test sheet with 3% MPC stems.

Two tests directed at qualifying a low usage level of monopotassium citrate (MPC) stems in RCB were already in progress at the BL Plant. These were a test sheet incorporating 2% MPC stems and a second test sheet made with 2% MPC stems + 2% citric acid (CA) stems. Test RCB containing 3% MPC stems will be made if subjective results are acceptable on the 2% MPC sheet.

Blended cigarettes containing RCB made with 11%, 16% and 22% citric acid stems have been made and submitted for analyticals. Subjective evaluation will concentrate on the RCB made with 11% CA stems based on the results of initial screening. If this sheet material is unacceptable, a lower level of CA stems in RCB will be tested (4-8%, depending on detectability of 11% CA stems).

Direct inclusion models containing 3% and 5% citric acid stems that were made into ES and IS at Louisville, as well as CA stems ammonia cased and Hauni tunnel treated in Semiworks, have cigarette analyticals pending. The preliminary screening of the ammonia treated material indicates an improvement over normal CA stems, but too much "dark" character (reacted ammonia/sugar flavors) when incorporated at 5% in a cigarette. The coarse CRS cut width now in use at Bermuda Hundred could preclude the direct inclusion option. The above IS and ammonia treated stems were also made into pilot RL to determine if these pretreatments increase utilization potential in sheet.

Park 500 trials have been requested to evaluate low level incorporation of citric acid stems in RL. Inclusion in RLTC is expected to have the highest probability of acceptance, and may be the best means of utilizing CA stems in brands not containing RCB.

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C. **Plans**: Produce plant test sheets to determine the maximum acceptable level of MPC stems and to qualify an initial usage level of CA stems. Produce pilot RL sheets to evaluate the subjective impact of CA stems used for double ART extractions.

II. LIQUID FLAVORS

A. **Objective**: Develop liquid flavor replacements for dry flavors in reconstituted sheet products.

B. **Results**: Takasago liquid flavor, compounded at the Flavor Center, was used to produce RLB test sheet (without dry flavors) at Park 500. The latest Chart liquid flavor sample met residual alcohol specifications, but had insufficient degree of roasting and was rejected from an analytical and subjective standpoint.

C. **Plans**: Evaluate Park 500 RLB with liquid flavors. Produce pilot RL-150B to evaluate new Chart flavor samples when available.

III. ASTA

A. **Objective:** Develop subjectively and physically acceptable reconstituted sheets for international application using cast sheet technology and proprietary binder systems.

B. **Results:** Analytical data on handmade 100% cigarettes comparing ASTA-II (pectin binder) handsheets to ASTA-I (DAP binder) handsheets showed that the pectin sheets gave a 40% reduction in static burn time and puff count. Tar, nicotine and TPM were reduced, but were higher on a per puff basis. Handsheets were made to define the interactive effects of tobacco particle size and pectin addition on ASTA-II sheet physical properties.

TSA forwarded the requested samples from the cast sheet facility in Cadiz, Spain. Two sets of dust blending silo discharge show consistent tobacco particle size (95% <120 mesh); chemical analyses are in progress. Finished sheet samples taken across a two day period during the Cadiz visit indicate product consistency via both TSA and PM-USA laboratory data. Similar samples from a subsequent five day operating period show uniformity by TSA analyses; these samples are also being analyzed at R&D.

C. **Plans:** Complete the initial evaluation of ASTA-II (pectin binder) handsheets. Complete the analysis of TSA samples to determine the analytical stability of the Cadiz process.

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PROJECT NUMBER: 1308
PROJECT TITLE: Papermaking Process Development
PROJECT LEADER: R. M. Rogers
PERIOD COVERED: November, 1989

I. HANDSHEET PRODUCTION

A. **Objective**: Develop proprietary cigarette papers for low sidestream and other new product applications.

B. **Results**: The latest samples of hydromagnesite, prepared in-house, were incorporated in low porosity sheets at 10% (+ 20% Multifex) to 30%. Commercial samples of this material typically resulted in porous sheets at a 30% inclusion level. The large agglomerates, observed in previous handsheets of 30% pyroaurite ($Mg_6Fe_2(OH)_{16}CO_3$) were eliminated by mixing the filler at high solids level (20%) prior to adding to the paper stock. This mixing technique will be used for future handsheets to improve filler distribution.

Samples of 1/4" cut cellulose acetate (CA) tow from two vendors (Eastman and Celanese) were evaluated for inclusion in web for cigarette filters (75% CA plus refined softwood). Both tow samples were uncrimped at approximately 2 denier. Only the Eastman sample dispersed easily and formed a uniform sheet with tensile, elongation, and porosity comparable to commercial filter webs. Eastman will produce 300 lbs of cut CA fiber for trials at Maine, tentatively scheduled for mid January, 1990.

Utilizing smoking results from the matrix evaluation, a model has been developed relating various properties of high weight papers (porosity, carbonate level and KH_2PO_4 at 63 gm/m^2) to static burn time and sidestream smoke. Burn rate is now being predetermined using this model.

A number of handsheet techniques are being investigated that apply and incorporate fiber/filler bands in a paper's structure. The objective of this evaluation is to reduce the burn rate of cigarettes in specific zones. Restricting the structural materials to fiber and calcium carbonate should minimize the subjective impact.

C. **Plans**: Continue the production of handsheets to evaluate experimental fillers and burn rate modifiers.

Obtain 300 lbs of cut cellulose acetate from Eastman for pilot trials at Maine.

Develop a process to incorporate structural bands of fiber and/or filler in cigarette paper handsheets.

II. PILOT PRODUCTION

A. **Objective**: Produce pilot quantities (bobbins) of cigarette paper.

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B. **Results:** The equipment required for the production of various papers on the RL pilot paper machine has been identified. Mass balance diagrams of both the RL and the University of Maine pilot units have been completed. Justification for this project is based on several aspects. In-house production guarantees confidentially, the RL machine is more suited to larger runs, and since the availability of Maine's facility may change, a defensive posture could prove advantageous.

Follow-up pilot trials at James River demonstrated that typical Marlboro paper (137-1) can be printed with sol-gel and processed through a commercial rotogravure press using a 200 lines/inch cylinder. This pattern applied 0.49 gm/m^2 of dry sol-gel (AlOOH). Since higher rates of addition severely weakened the sheet, greater amounts must be applied with multiple passes.

The precipitated boehmite (AlOOH) produced at Lee Labs is being washed to reduce its chloride concentration to a level acceptable for paper production. A computer simulation of a typical paper machine indicates that a 95% decrease in chloride reduces a machine's whitewater chloride level below 15 ppm and sheet concentration to a level well below 90 ppm. Since commercial papers typically contain 170 $\pm$ 120 ppm chloride, the projected contribution of chloride from the AlOOH should not pose a problem.

Kimberly-Clark completed a series of four high basis weight papers at 63 gm/m^2 to identify the optimum porosity and calcium carbonate level for this model. These sheets were produced under a proprietary agreement with Philip Morris covering high basis weight papers. The papers will be sized by PM to determine the optimum level of KH_2PO_4 required.

A literature search by TIS revealed general information and a few patents for the utilization of tobacco fibers in cigarette papers. Some of these products were actually developed past the pilot stage but large scale commercial production was not achieved. All the processes modified conventional wood/nonwood pulping technology to separate the individual fibers, including screening to remove the undesirable fine fraction. It does not appear that the concept of including tobacco fibers in cigarette papers is proprietary.

C. **Plans:** Complete production of scheduled papers at University of Maine.

Evaluate the line contrast of the polished vergeure roll at Maine.

Issue specific recommendations on the equipment required for production of various papers on the RL machine.

Maine will prepare a proposal to identify the comparative quality of tobacco fibers versus flax fibers.

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PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: November 1989

I. BINDER TECHNOLOGY

A. **Objective**: Investigate the mechanism of filler bonding and stiffness produced by coating. Improve commercial feasibility by examining alternate binders and processes.

B. **Results**: Firmness data from cigarettes bonded with alternate binders, potato maltodextrin and potato starch were received and analyzed. The control pectin coated filler cigarettes were firmer than the cigarettes produced with potato maltodextrin and potato starch coated filler. Further work would be required to determine if modification of the processing parameters on the low density rod maker could enhance firmness with these binders. The cigarettes will be tested for subjectives.

C. **Plans**: A completion report will be written on alternate binders.

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PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: November, 1989

I. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

A. **Objective**: To conduct trials providing information for development of the ART commercial process.

B. **Results**: Results from the test conducted at Bermuda Hundred processing ART filler at various tunnel operating conditions were reviewed with BH personnel. (No effect on filler sieve size was demonstrated. This was contrary to previous results from Semiworks testing that had shown significantly more breakage at a steam pressure of 8 bar versus 4 bar.) A decision was made to run additional tests at BH. In addition, a test series was initiated at the Semiworks to evaluate the effect of steam pressure/tobacco bed depth on degradation. The first test, with Marlboro cut rag, was run and again showed that the filler processed at 8 bar had a >6+12 mesh value that was 10% less than runs processed at lower pressures. A run with ART filler is scheduled.

Two Semiworks trials were run casing DL strip with ammonium hydroxide in an effort to achieve the same pH adjustment as with ammonium bicarbonate but at a lower moisture level. Chemical results showed that the target value of ammonia addition (0.54%) was not reached, as the trials achieved levels of 0.11 and 0.28%.

In the clay-mediated separation program, a simplified fluidized bed test model was constructed for pressure drop and flowrate evaluations. The results will be applied to the design of the laboratory separation unit being built for Chemical Research. Tests with the coarse frit (25-50 micron maximum port size) and the medium frit (10-20 micron) have been conducted using the standard Clarion 470 clay. A new sample of clay was received from American Colloid, which is quoted as containing no recycled fines and thus has a larger mean particle size.

C. **Plans**: Complete test series determining the effect of steam pressure on ART filler physical properties.

Conduct investigations into achieving the required pH adjustment for ART filler at a lower moisture level. This may include processing changes or use of alternate bases.

Continue laboratory experimentation in the clay-mediated separation program.

II. HAUNI HT TUNNEL EVALUATION

A. **Objective**: Determine the effect of steam conditioning tobacco materials in the Hauni HT steam tunnel prior to drying.

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B. **Results**: Final subjective evaluation (after aging) of the test series evaluating various post tunnel treatments indicated that all conditions (countercurrent, cocurrent, and cooled to ambient conditions prior to countercurrent drying) were different from the non-tunnel treated control. The last test condition had not been different from the control in the initial smoking tests.

Spotting results were received from the study to evaluate the effects of tunnel processing of DIET feedstock. Cigarettes containing 100% and 12% (Marlboro-type blends) of the following test materials were evaluated on the SOLVIC unit (5 days aging): MC DIET, D Pilot Plant DIET, and D Pilot Plant DIET that was treated in the tunnel prior to expansion. All cigarette groups showed 1 critical spot/2000 cig, indicating that the tunnel treatment did not reduce the spotting level. However, this value is significantly lower than those yielded from preliminary tests which had shown tunnel treated material to have lower spotting levels in 100% models (45 critical spots/2000 cig reduced to 28 and 32 reduced to 7). Cigarette firmness results indicated a loss of firmness with tunnel processing. In 100% DIET cigarettes and at a constant tobacco weight, the firmness values were 2.9 mm for the D Pilot DIET, 3.1 mm for the D Pilot DIET with tunnel treatment, and 3.2 for MC DIET.

Semiworks trials were run to evaluate the effect of tobacco moisture (21 and 25% OV) on Hauni treatment and to compare the performance of a steaming cylinder versus the tunnel. In the latter trial, process measurements indicated that the filler processed through the cylinder reached a temperature of 160 F with a residence time of 20 seconds. Firmness testing indicated that increasing the moisture from 21 to 25% yielded a slight advantage and that the cylinder produced about half of the improvement of the tunnel. Firmness values at a constant tobacco weight were:

Control	3.4 mm
HT @ 21% OV	2.8 mm
HT @ 25% OV	2.7 mm
Cylinder (21% OV)	3.1 mm

C. **Plans**: Due to the low spotting values, conduct a replicate test evaluating the effect of tunnel processing on DIET feedstock.

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PROJECT NUMBER: 1810
PROJECT TITLE: ART Process Development
PROJECT LEADER: R. Prasad
PERIOD COVERED: November, 1989

I. PROJECT ART - COMMERCIAL PLANT SUPPORT

A. **Objective**: To support the Bermuda Hundred Processing Facility startup/operation.

B. **Results**: Support to the commercial plant process optimization continues to be provided. Testing of modified CO_2 fill procedure to minimize bed upsets and CO_2 density fluctuations was initiated at the Commercial Plant. CO_2 flow rates as high as 1400 lbs/min were achieved compared to current flow rate of 1000 lbs/min. Further testing is in progress.

Double batching of stems was tested at the Commercial Plant, i.e., two batches of filler were extracted with a single batch of stems. Extraction targets were met by increasing the amount of stem in the absorber from 1800 lbs dwb to 2100 lbs dwb for the double-batch operation.

Ongoing work at the University of Texas and Pemm Corp, New York, shows the importance of bed packing uniformity and the bed upsets that can occur with fast CO_2 fill and ramp rates. In addition, the design of CO_2 inlet flow distribution system is key in achieving high flow rates without "rat hole" formation or bed bypassing. One-third of the extractor pressure drop should be across the bottom screen to assure uniformity of CO_2 flow distribution. This will require the open area at the bottom screen to be reduced from the current 30% open to about 0.2-1.0% open area. This design change was recommended to the plant management.

C. **Plans**: Continue to support the commercial plant optimization.

II. PROJECT ART - PILOT PLANT

A. **Objective**: To support commercial plant design and flavor development objectives at the Bermuda Hundred Pilot Plant.

B. **Results**: A series of tests at reduced stem OV and/or CPI of CRS were successfully completed to reduce the pressure drop of the absorber. Significant increase in CO_2 flow rate at reduced pressure drop were obtained via reduction of stem OV from 38% OV to 25% OV and/or reduction in stem cuts/inch from 150 cpi to 53 cpi. A combination of stems cuts/inch and OV reduction was implemented at the Commercial Plant, i.e., 76 cpi and 30% OV. Tests are scheduled to increase CO_2 flow rate to design target conditions.

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Filler OV reduction (from 28% OV to 22% OV) was successfully tested to reduce the extractor pressure drop and to achieve higher CO_2 flow rates. However, the filler was underextracted due to the low AB level used. Development of ways to apply ammonia and/or alternative bases has been initiated with a target filler OV of 21%.

Batch water column trials were started with appropriate safety protocols in place. Initial testing with acid water was completed (without filler) to determine appropriate operating conditions. A CO_2 flow rate of 267 lbs/min resulted in no carry-over of acid water solution from the absorber into the pipes. A run with pre-extracted filler was completed to simulate full operating conditions. Training of operating personnel in full safety gear was done, along with sampling of nicotine in air. As expected, the filler increased in moisture content from 25% OV to about 30-35% OV. The nicotine content of pre-extracted DL blend filler was reduced from 0.15% to 0.09% dwb using acid water in the baskets.

The first batch water column run was successfully completed. DL blend filler was extracted from 2.73% nicotine to 0.07% nicotine level (97.3% extraction) at 2000 psi, 140°F, 200 M/M. No carryover of acid water from the absorber to the pipes took place at 267 lbs/min CO_2 flow rate. However, due to an unexpected "stop cycle" during CO_2 recovery step, backflow of acid water to the lower baskets was experienced. The nicotine from CO_2 was absorbed in the bottom two baskets, indicating excellent entrapment capabilities of acid water as well as excellent efficiency of contact between CO_2 and water provided by the basket sieve tray design. As expected, the filler OV increased (from 25.8% initially to 28.8% "fluffed").

Loss of non-nicotine volatiles, "flavors", was measured by GC in the water effluent from the batch absorber. Total loss of volatile flavor, compared to the amount of nicotine removed, was in the range of 0.02 to 0.07 parts per one part nicotine. Subjective testing of extracted filler is in progress.

A safety review of pilot plant procedures was completed to determine the feasibility of running DI water in the batch water column. The required safety protocols to protect from nicotine in air were outlined. A limited number of runs with DI water are possible at BHPP provided the new safety protocols are followed. The expected filler subjectives should be equivalent to the proposed interim water column "pipe". This negates the need for a 12 inch pipe at an additional cost of $250M to $500M in the 650 being prepared by PM Engineering.

C. **Plans**: Test the extractor pressure drop reduction at reduced OV and at adequate AB level. Implement the finding in the Commercial Plant to achieve capacity increase.

Explore process alternatives to improve the product attributes.

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III. PROJECT ART - NEW PROCESS DEVELOPMENT

A. **Objective**: To develop new processes for scale-up and implementation in the Bermuda Hundred Facility.

B. **Results**: A preliminary scope of separations pilot plant using clay systems was prepared using fluidized bed concept in the upflow mode of operation. Further discussions with designers from clay equipment vendors indicated that upflow fluidized bed design is not practiced in the industry and a plate and frame filter is the preferred option.

A literature survey of the clay applications in the industry was initiated. The measurement of key process parameters for the design of pilot scale system was initiated, i.e., settling velocity, fluidization velocity, particle size distribution vs pressure drop.

A 650 for continuous water column is being prepared by PM Engineering. Plans are to install the cooling heat exchanger in February 1990 and to install the continuous water column by September 1990.

C. **Plans**: Continue laboratory and pilot scale development activity on selective separation of nicotine from flavor.

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PROJECT NUMBER: 1811
PROJECT TITLE: Process Chemistry Development
PROJECT LEADER: S. E. Wrenn
PERIOD COVERED: November, 1989

I. PROJECT ART - pH ADJUSTMENT OF PRE-EXTRACTED FILLER

A. **Objective**: To replace AB with a suitable agent which would meet chemical, subjective and processing qualifications as a pre-treatment of ART filler.

B. **Results**: Trials were made in Semi-works to replace the AB with ammonium hydroxide as a pre-treatment on ART strip. Because ammonia has a higher solubility than AB, the needed base could be added and result in an OV of <21%. ART strip at 16% OV was sprayed with ammonium hydroxide solution, however, the majority of ammonia was not retained on the tobacco.

C. **Plans**: Other alternatives to AB are being investigated in the laboratory.

II. PROJECT ART - NEW BLENDS

A. **Objective:** To characterize new ART blends and blend components by titration analysis.

B. **Results:** Twenty-four hour titration analysis of various ART blends and blend components were performed to determine buffer capacities which can be directly related to extractability. Titration of the blends indicated that an increase in buffer capacity was related to the increase in burley content.

C. **Plans:** Evaluate any new strip blends used in the ART process. Use the information from the titration curves to predict performance of alternate bases. Evaluate stem material by titration analysis.

III. MENTHOL GLUCOSE CARBONATE

A. **Objective:** To formulate menthol glucose carbonate (MGC)/solvent system with desirable physical and chemical characteristics to allow direct application to a cigarette rod.

B. **Results:** A new batch of MGC was produced at Lee Labs in Petersburg under direction of Bill Edwards (Chemical Research). Samples from various process stages will be evaluated for direct application onto a cigarette rod.

C. **Plans:** Analyze samples of MGC from the various distillations stages for physical properties.

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PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: L. Ryan
PERIOD COVERED: November, 1989

I. METHOPRENE

A. Objective: To determine the most efficient use of the insect growth regulator, methoprene, within PM's cigarette beetle (CB) management program.

B. Results: Greater than 93% control of CBs was achieved by spraying 500 ppm of methoprene onto the inner surface of cardboard cases used for export (1). This level of control is an improvement on similar studies using oriental bales (2) but did not achieve the 100% control of CBs achieved by direct methoprene application to lamina (3).

When methoprene-treated lamina was run in the DIET process, the methoprene retained its biological activity and resulted in 100% CB control (4).

C. Plans: Continue to assist in the integration of methoprene into PM's pest management program.

D. References:

1. Lehman, R. Control of the Cigarette Beetle in Tobacco Stored in Methoprene-treated Cases. Special Report No. 89-050.

2. Faustini, D. L., Lehman, R.M., Long, J.S. and Manzelli, M.A. The Efficacy of Kabat® as a Surface Spray on Oriental Tobacco Bales for Control of the Cigarette Beetle Lasioderma serricorne (F.) (Coleoptera:Anobiidae). Beitrage zur Tabakforschung International, 14(1): 41-44, 1987.

3. Manzelli, M. A. Management of Stored-Tobacco Pests, the Cigarette Beetle (Coleoptera:Anobiidae) and Tobacco Moth (Lepidoptera:Pyralidae) with Methoprene. Journal of Economic Entomology, 75(4): 721-723, 1982.

4. Ryan, L. Protecting ET in Storage with Methoprene - Biological Results. Memo to Faustini, D. L. October 7, 1989.

II. SERVICE TO OTHERS

A. Objective: To provide technical services to areas outside R&D.

B. Results: Assistance was given to Engineering Department personnel in their project to apply methoprene (Kabat®) to all expanded tobacco (1). Cigarette beetles were supplied to QA personnel in PM

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Europe for steam cylinder efficacy tests (2). Advice was given to the PM affiliate in Coast Rica regarding infestation control.

C. **Plans**: Continue to provide technical assistance as requested.

D. **References**:

1. Ryan, L. Cylinder Spraying of Kabat® - Methoprene Residue Results. Memo to Sweeney, W. R. November 9, 1989.

2. Ryan, L. Request for Cigarette Beetles. Memo to Freymond, J.-M. October 12, 1989.

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PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: November, 1989

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. Objective: To develop methods to objectively and reliably evaluate human responses to smoke constituents and tobacco flavorants.

B. Results:

Cognitive NERP Study

Cognitive NERP experiments aimed at evaluating two alternative natural menthol sources (source one and source two) as compared to the current source (control) continue. To date, eight subjects have been presented each comparison.[1] The data from each subject are being analyzed *via* step-wise discriminant analysis. Preliminary analyses indicate that two of eight subjects could discriminate between the menthol control and the menthol from source one and five of eight subjects could discriminate between the menthol control and the menthol from source two. These results are consistent with subjective tests conducted on these samples and indicate that source one may be a better substitute for the current menthol source than source two.

C. Plans: Complete testing of ten subjects with each alternative *vs* current menthol comparison and continue to employ step-wise discriminant analyses to analyze the cognitive NERP data from *individual* subjects. Following the analysis of all of the data, plans are to determine how the degree of discriminability between these menthols relates to the other menthol discriminations that have been tested.

D. Reference:

1. Martin, B. Notebook No. 8846, pp. 71-80.

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PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: November, 1989

I. PACK INSPECTION SYSTEM

A. Objective: Develop and implement a method for the on-line inspection of cigarette packs.

B. Results: The reset circuit required for the laboratory pack inspection system has been completely debugged. The reset circuit provides the synchronizing signals for both the imaging camera and the Androx imaging board.

The laboratory pack inspection system was transferred to Edwards Machine Co. in Ashland, Va., for testing on a vacuum conveyor designed by PM Engineering. The primary objectives of the test were to verify the reliability of the image acquisition components and to verify that the decision to reject a pack could be reached before the pack passed the reject station. Images of a large number of Parliament packs were also acquired for further testing of the discrimination capability of the system.

Software development for the laboratory pack inspection system has continued. The software has been developed to allow extensive flexibility in the choice of inspection options and to enable the calculation of the statistics needed to quantify the performance of the system in the upcoming factory trial.

In collaboration with Development Engineering, the hardware specifications are being developed for a factory prototype inspection system. Unlike the current laboratory inspection system, the factory prototype will be suitable for long-term installation in a factory environment and will have the capability for inspecting three views at the rate of 15 units per second. Several vendors have been identified with the capability for providing the image acquisition and analysis hardware needed for the factory prototype. Discussions with each of these vendors are ongoing.

The pack inspection system carries out its inspection on binary images obtained by thresholding gray scale images. A method for automatically determining the optimum threshold continues to be an area of active research. Simulations have shown that a correlation criteria for threshold selection works well for direct images but does not work well for edge detected images. Current research is directed toward generalizing the correlation criteria so that it works equally well in both cases.

C. Plans: Carry out a factory trial of the PM pack inspection at Stockton Street. Complete the hardware specifications for the factory prototype.

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II. INDIVIDUAL CIGARETTE INSPECTION

A. **Objective**: Develop a method for high resolution, on-line inspection of individual cigarettes.

B. **Results**: Optical hardware for the fully focused acoustooptic (AO) scanner was set up and tested. The AO cell was used in a negative focal length mode. A spot of about 0.4 mm diameter was obtained with a scan rate exceeding 100,000 scan/second. Additional work is now in progress to provide a full cigarette length scan with better control of the distance between the cigarette and the scanner.

III. AEROSOL GENERATION AND MEASUREMENT

A. **Objective**: Develop and characterize a laboratory aerosol generator capable of producing highly concentrated aerosols.

B. **Results**: A method has been devised to measure the aerosol concentration exiting the generator using an absolute filter and a 500 ml syringe. Filter weights have been found to be stable even when loaded with glycerin. The method has been used to quantify the losses in the generator due to vapor condensation on the walls of the cooling section. Vapor losses of approximately 50% have been observed.

A commercial ultrasonic aerosol generator has been evaluated. The generator is capable of producing a dense water aerosol but is ineffective in producing an aerosol from pure glycerin. A solution of 30% glycerin in water was successfully nebulized.

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PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: November, 1989

I. LOW NICOTINE

A. **Objective**: Support to ART Commercial Plant.

B. **Results**: The CO2 - tobacco hydraulic tests scheduled at The University of Texas have been completed. Twelve runs were conducted to study the effects of tobacco OV, packing density, fluid velocity ramp rate, bed freeboard, and CO2 fill procedure on "rat hole" formation and pressure drop across the filler bed. As expected reducing filler OV and decreasing the bed packing density had the greatest effect on reducing pressure drop at a given flow rate. No definite conclusions could be reached on how flow rate influenced pressure drop because of wall effects from the three inch column. Other observations made during the experiments are shown below.

1) The tobacco moved as a "slug" through the column. Typically the bed lifted when the CO2 velocity was between 3.5 to 4.5 ft/min. Generally the bed split at less than a foot from the bottom.

2) Tobacco did not float in liquid CO2.

3) No entrainment of tobacco particles was seen to occur even at fluid velocities of 4 to 5 ft/min.

4) Filling the system with liquid CO2 during start-up caused "bumping" of the tobacco bed which did not occur with hot gas filling.

C. **Plans**: The experiments were recorded on video and presented to management. A report has been issued.

II. LOW NICOTINE

A. **Objective**: Support ART Commercial Plant corrosion studies.

B. **Results**: A Dewluxe 20 Moisture Analyzer (Stephens Equipment Co.) was evaluated for measuring the amount of solubilized water in CO2 at process conditions. Several adjustments of sampling conditions, sampling hardware, and standard amounts did not yield reproducible data. It was recommended that the test be discontinued and the instrument returned to the vendor as not being suitable for our application.

C. **Plans**: Continue to investigate other methods and equipment for moisture analysis in SC-CO2.

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III. LOW NICOTINE

A. **Objective**: Develop second generation processing for ART.

B. **Results**: At the request of The Analytical Research Division runs were made on the one-liter liquid absorber system to obtain scrubber solutions of pure water and 4% monopotassium citrate. The solutions will be analyzed for differences in SC-CO2 soluble tobacco components, other than nicotine, which may partition into the liquid phase. The runs were made at current ART conditions using DL5 filler.

C. **Plans**: Work is on-going.

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PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: November, 1989

I. **OPERATIONS SUPPORT** (J. Crump in collaboration with the Applied Technology Group)

A. **Objective**: Determine the rate and consistency of sideseam adhesive application during manufacturing.

B. **Results**: A study has been initiated to determine the quantity of sideseam adhesive applied to cigarette paper as well as the rate of adhesive delivery. Measurements are currently being made on a Mark 9 maker in Bay 4 at the MC.

II. **PROJECT ART (NICOTINE STUDIES)** (A. K. Basak)

A. **Objective**: Characterize the behavior of nicotine in aqueous solution.

B. **Results:** Work has been initiated to determine the effect of ionic strength on the acidity constants of nicotine. NMR experiments are in progress with the objective of elucidating the unusual temperature dependence observed for the acidity constants of nicotine. Results are not yet available.

III. **MENTHOL STUDIES** (T. V. Van Auken)

A. **Objective**: Determine the mechanism of menthol migration.

B. **Results**: Experiments were conducted to distinguish between two possible mechanism of menthol migration: a) Vapor phase migration, or b) Migration via contact and capillary action. The results show conclusively that menthol migrates via the vapor phase. No further work is planned.

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PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: November, 1989

I. REDUCED SIDESTREAM/PAPER TECHNOLOGY (YI, SANDERS)

A. Objective: Examine the ultrastructure of selected cigarette papers and paper additives in support of the low sidestream project.

B. Results:

Five paper, char, and ash samples from $45g/M^2$ basis weight papers were examined and compared. Each paper contained a different K containing fluxing agent. The ash from papers with KH_2PO_4, K_2HPO_4, and K_3PO_4 (2.9% K) remained intact unlike the $18g/M^2$ basis weight papers. The lighter weight papers with the same additives disintegrated after they were burned[1]. The ash from the heavier weight papers with the three fluxing agents mentioned above as well as papers with KCl or K_2 acetate fluxing agents, formed ash that were light to dark gray in color. With additional burning or with increased temperatures, however, the ash turned white in color. Paper with KCl burned faster than the four other papers. The ash and char from all five papers exhibited a glazing effect and/or fused crystals[2].

Two papers were examined that were coated with precipitated sol gel produced at Lee Labs. The sol gel was divided into two batches and washed to remove chloride ions, one by centrifugation, the other by membrane filtration. One paper was coated with the centrifuged washed sol gel and the other paper was coated with the membrane washed sol gel. The two washing procedures made no difference in the way the sol gels coated the flax fibers of the cigarette papers[3].

Two hydromagnesite samples ($Mg(CO_3)_4 \cdot 4\text{-}5H_2O$) were examined. One sample was prepared using $Mg(NO_3)_2$ while the other was prepared using Mg acetate. Both samples consisted of aggregates of thin platelets with curled edges[4].

Paper, ash, and char from two Japanese-made cigarettes were examined. Both the Mild Seven and the Marlboro cigarette papers contained Ti along with Ca. The Mild Seven ash maintained its paper shape and exhibited a smooth glazed surface with interconnected particles. The Marlboro from Japan burned similarly to the U.S. Marlboro. Its ash was fluffy and fell apart after being burned[5].

C. References:

1. Yi, G., "Structural Characterization of Two Inner Wraps Containing K_2HPO_4 and K_3PO_4," Memo to S. Tafur, September 28, 1989.
2. Yi, G., "Morphological Characterizations and Comparisons of Experimental Cigarette Papers," Memo to S. Baldwin, October 30, 1989.

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3. Sanders, K., "Examination of Papers XHIL-4 and XHGA-3," Memo to B. Edwards, October 30, 1989.
4. Yi, G., "Structural Comparisons of Two Hydromagnesites," Memo to J. Fournier, October 29, 1989.
5. Yi, G., "Structural Analysis and Elemental Contents of Mild Seven Lights and Marlboro King Produced in Japan," November 4, 1989.

II. SUPPORT TO OPERATIONS (SANDERS)

A. **Objective**: Provide analytical support to R&D.

B. **Results**: Two Gelman TPM pads were examined for structural differences but were found to be the same[1].

C. **References**:

Sanders, K., "Analytical Microscopy Result Form," to J. Lewis, October 23, 1989.

III. MISCELLANEOUS (BALIGA, SANDERS)

A Malvern particle size analyser was ordered and received. It can measure and plot the size distribution of particles in solution from 0.1µm to 500µm in diameter. This will be used to measure the size distribution of production batches of precipitated sol gels.

The article, "Children of Alcoholics," by Karen Sanders, published in the November 1988 issue of the Virginia Lifeline journal, was awarded 2nd place Feature Article by the Virginia Publishers wing at the State of Virginia VAVRS convention.

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PROJECT NUMBER: 1752
PROJECT TITLE: Molecular Structure Determination
PROJECT LEADER: G. Vilcins
PERIOD COVERED: November, 1989

I. CARBON 13 NMR ANALYSIS OF NICOTINE ON CLAY

A. **Objective**: To determine the degree of loading of nicotine in the clay and the type of chemical species present.

B. **Results**: A nicotine saturated clay was extracted to remove the nicotine. The residual nicotine in the clay was detected by ^{13}C CPMAS NMR. The signals were broadened by paramagnetic iron atoms in the clay structure. By measuring the signal intensity as a function of the cross polarization contact time, the ^{1}H rotating frame relaxation rates were measured. The relaxation appeared to be non-exponential either because of the suppression of proton spin diffusion due to the paramagnetic centers or because there are two or more nicotine sites in the clay with intrinsically different relaxation rates. By assuming two nicotine sites with different rotating frame relaxation rates, a reasonable approximation of the relaxation curves were obtained. The relative proportion of the two types of nicotine in the extracted clay differed from a saturated clay sample but the relaxation rates appeared to be the same.

C. **Conclusions**: Nicotine adsorbed in clay may be distributed between two different sites. Extraction of the nicotine caused a redistribution of the residual nicotine between the two sites. This suggested a stronger affinity for one site over the other.

D. **References**:

Wooten, J., "^{13}C CPMAS NMR of Nicotine Adsorbed on Clay," memo to J. Seeman, November 1, 1989.

II. WATER COLUMN STUDIES FOR THE ART PROCESS

A. **Objective**: The effect of monopotassium citrate in the aqueous extraction of carbon dioxide tobacco solubles was determined.

B. **Results**: The SFE/SFC instrument was configured to perform online extraction and gas chromatographic separation. Nicotine extracted in the carbon dioxide was monitored by ultraviolet detection at 274 nm. The effluent was passed through a restrictor into a heated split injector where it was cold trapped on the front end of a capillary column. Chromatograms were obtained of each tobacco extraction performed with no water, water, and 6% monopotassium citrate. Peaks were identified by GC/FT-IR/MS.

C. **Conclusions**: Final evaluation of the data is in progress.

D. **References**:

Shafer, K., PM Notebook #8789 pp. 55.

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PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: November, 1989

ANALYTICAL FLAVOR SPECIFICATIONS

A. **Objective**: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities. To provide analytical certification on export flavors manufactured at the Flavor Center for FRG compliance.

B. **Results**:

Contaminant analyses continue on tobacco flavors, revisions of processed oils, aroma chemicals and possible vendor alternates for specific materials. All codes processed by the Flavor Center have now been completed. Approximately 30 remaining materials will be obtained from the specific usage sites for contaminant analysis.

Certification for the PMI samples continues and two specific analyses are no longer required.

A modified head space sampling procedure was evaluated for use in the certification process by the Dionex equipment. Various vial/septa combinations, sample matrix changes and heating times were examined. Alternatives to these procedures are being investigated.

Samples received from K. Podraza and H. Secor were evaluated for nicotinic acid content. Additional components were examined by a variety of GC, LC and GC/MS techniques.

Round robin participation for Flavor Center QA was conducted to assist in the sugar analysis procedure refinement.

Training of QA specialist on the FRG methodology and basic instrument operation, spectral interpretation, GC and HPLC methodology continues.

Two members participated in a special smoke panel group.

C. **Plans**: Continue the certification of the PMI samples, begin the certification of all flavors currently on hand at the Flavor Center and continue the specification work for the second vendor.

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PROJECT NUMBER: 1759
PROJECT TITLE: Materials Evaluation & Elemental Analysis
PROJECT LEADER: P. F. Grantham
PERIOD COVERED: November, 1989

I. MATERIALS EVALUATION

A. **Objective**: To identify components of commercial products prior to their use at PM facilities.

B. **Results**:

No tours or presentations were made this month. Routine analysis of commercial products, Packaging Development, and TTG samples continue.

At the request of Miller, polymeric coatings on the inside of Miller and Coors beer cans were analyzed using FTIR to determine differences in composition. The coatings were all composed of an epoxy/styrene copolymer and an acrylic polymer. Coors appears to use a higher percentage of the acrylic polymer than does Miller. We are awaiting microscopy data on coating thickness.

C. **Plans**: A tour of the Louisville facility is scheduled for November 28 and 29. Presentations will be made as requested.

II. ELEMENTAL ANALYSIS

A. **Objective**: To provide qualitative and quantitative elemental data on tobacco, cigarette paper, development and materials evaluation samples.

B. **Results**:

Analysis of paper samples and tobacco using XRF and atomic absorption methods continued.

An assay was developed to determine trace chloride impurities in menthylchloroformate using a selective ion electrode. Comparisons were made with PPG (the manufacturer) and Lee Laboratories, who uses the compound to produce glucosementhyl carbonate for Philip Morris. The comparison between the three laboratories was excellent. No further samples are anticipated, since the suppliers can now do their own analysis.

Thermogravametic Analysis (TGA) was performed on four tipping paper samples suspected to cause filter flare up. Three cork papers and a white base sheet were analyzed to determine their thermal decomposition characteristics. The three cork papers showed a decomposition at 250°C. The white base paper did not show a sharp decomposition at this temperature. This would indicate that the ink or fillers in the ink system cause the decomposition temperature to decrease.

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C. **Plans**: Support to other R&D projects for elemental analysis will continue. Methods for the determination of trace metals in glucosementhyl carbonate will be developed. Five filler materials for the cork tipping papers will be analyzed using TGA.

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PROJECT NUMBER: 1902
PROJECT TITLE: Cell/Tissue Culture Research
PROJECT LEADER: W. P. Hempfling
WRITTEN BY: D. M. Teng
PERIOD COVERED: November, 1989

I. TOBACCO MICROBIOLOGY

A. **Objective**: To develop methods and to evaluate the microflora resident in tobacco materials.

B. **Results**:

1. "Famous" Blend Storage Study

After sixteen weeks of storage, bacterial populations measured in the "Famous" blend stored in the warehouse and desert room were not significantly different from populations in the twelve-week samples. Mold and yeast populations were less than fifty colony-forming units (CFU) per gram of tobacco.

2. Dry Flavor Study

This study was initiated to determine the microbiological characteristics of dry flavor ingredients before and after their use in the formulation of finished flavor systems. To date, five sampling collections at each location have been completed at the MC Flavor Kitchen and Stockton Street Factory. The magnitudes of bacterial and mold populations were similar in the samples from these two facilities.

3. Bulk Tobacco Handling Program

The evaluation of microbial populations in nine independent samples of different tobacco materials in the "rolling silo" was completed. A memorandum will be prepared describing the results.

4. Tobacco Storage Study

As a part of a three-year study designed to determine the effect of storage in different containers (hogshead, box and bale) on properties of stored tobacco strip, the microbial populations of the 1986 crop year bright strip were measured.

To evaluate various box liners, tobacco samples from bright (1989 crop year) were analyzed for their contents of bacteria, mold and yeast. These two components of the Tobacco Storage Study are ongoing.

5. Centrifuge Sludge Study

A study was initiated to determine the microbial populations of centrifuge sludge from the Park 500 Facility.

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C. **Plans**:

1. Continue the "Famous" blend storage study for an additional eight weeks.

2. Initiate the Dry Flavor Study at Park 500.

3. Continue the Tobacco Storage Study.

4. Complete the Centrifuge Sludge Study in December 1989.

D. **References**:

1. Chadick, D. Notebook No. 8825, pp. 100, 103-107.

2. Gaines, O. Notebook No. 8690, p. 199; Notebook No. 8904, pp. 2,6.

3. Jones, J. Notebook No. 8590, pp. 118-122.

II. ALTERNATIVE PRESERVATIVE PROGRAM

A. **Objective**: To develop procedures and to conduct microbiological screens to evaluate nature-identical preservatives as replacements for, or adjuncts to, propylparaben.

B. **Results**:

1. Phase I Screens

The following compounds were evaluated for the antimicrobial activity in the Phase I agar inclusion assay: tiglic acid, cinnamic acid, linalool, anisole, anethole, p-hydroxycinnamic acid, p-hydroxyanisole, ethyl anthranilate, levulinic acid, diethyl-l-tartrate, 4-carvomenthenol and 3-methylcrotonic acid. None of these compounds displayed growth-inhibiting activity equal to that of propylparaben at the level of 500 µg/ml.

2. Phase III Screens

The efficacy of thymol as a preservative for SEL was evaluated in the Phase III fermentor screen at concentrations of 150, 300, 450 and 650 ppm (compared to a thymol-free control). Preliminary results indicated that at 600 ppm, the pH change (an indicator of spoilage) in the SEL was minimized for up to 48 hr. When thymol (450 ppm) was added to the SEL at 55°C, and maintained at that temperature for two hours before lowering the temperature to 42°C for the rest of the experiment, the pH change was minimized for up to 48 hours.

3. Studies of Chemical Indicators of Spoilage (HPLC)

An additional amount of a compound in "spoiled" SEL with a retention time of ca. 23.85 min was isolated. The compound was

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quantitatively extracted into diethyl ether, and the extract was submitted to the ARD personnel for identification.

A second HPLC apparatus was assembled with a differential refractive index detector for the estimation of sugars in SEL.

A polymer-based HPLC column, capable of separating standard sugars, was inactivated when a dilute SEL solution was applied.

4. Multipurpose Bioanalyzer® Study

The Multipurpose Bioanalyzer® (Provesta Corp.) with the lactic oxidase-oxygen electrode was examined as an alternative method for assay of lactic acid in SEL. This apparatus may be suitable for on-line application at the Park 500 Facility. Examination of SEL samples taken from a fermentor at various times after initiation of spoilage showed that lactic acid increased and then decreased with time as expected from previous results. SEL, itself, interfered with electrode performance, and deposited residue on the electrode membrane so that the membrane required replacement.

C. **Plans**:

1. Continue the Phase I screening of prospective preservatives.

2. Evaluate mixtures of thymol and β-cyclocitrylidene acetic acid in the Phase III screening procedure.

3. Examine other types of HPLC columns capable of separating sugars in the presence of SEL.

4. Seek remedies for the interference of SEL in the assay of lactic acid with the Multipurpose Bioanalyzer®.

D. **References**:

1. Tenhet, S. Notebook No. 8281, pp. 149-154.
2. Teng, D. Notebook No. 8788, pp. 72-77.
3. Weissbecker, L. Notebook No. 8822, p. 67.
4. Shulleeta, M. Notebook No. 8404, pp. 86-89.

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PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: D. J. Ayers
WRITTEN BY: V. S. Malik
PERIOD COVERED: November, 1989

I. LOW NICOTINE STUDY

A. **Objective**: To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) level and specifically to isolate PMT from tobacco root extracts.

B. **Status**: The roots from Group 21 hydroponically grown Burley 21 tobacco plants were harvested and stored at -80°C. These plants were harvested 3 days after topping for maximum PMT activity as determined by a PMT time course study previously completed (1). All tobacco roots from Group 19 Time Course Study and Group 20 harvested 3 days after topping have been processed through the ammonium sulfate stage. Specific activities for group 20 plants ranged from 32.8 to 40.0 nmoles of N-methylputrescine formed per mg of protein in 30 minutes at 30°C (2,3).

Ammonium sulfate preparations were fractionated on two phenyl-Sepharose columns. Active fractions were pooled, dialyzed and frozen in aliquots at -80°C. The specific enzyme activities of these preparations represent about a 6-fold increase as compared to the ammonium sulfate samples (3).

A BioGel column was prepared to remove the low molecular weight contaminants from PMT samples. The standard compounds used for calibration were bovine serum albumin, ovalbumin, chymotrypsinogen and cytochrome c. A linear relationship existed between the log MW and the elution volume for the standards; however, the void volume of the column could not be determined accurately (4).

Isoelectric focusing (IEF) of PMT samples using the ServaNet IEF system may be useful for further identification of PMT. Some PMT activity has been recovered using the ServaNet system. The more desirable pH range gels (pH 4-6) requires further development (5).

Studies to examine putrescine-PMT binding were initiated. PMT could not be efficiently eluted from the DEAE-Sepharose matrix using 0.66 mM putrescine.

Ammonium sulfate and phenyl-Sepharose samples from the time course study were adjusted to 2.0 mg protein/ml for submission to Protein Databases, Inc. (PDI). Another aliquot of PMT sample eluted with putrescine from a DEAE-Sepharose column was processed for PDI by washing with water and 1X PDI buffer using the Centricon 30 (Amicon) microconcentrating unit. Before submission to PDI, samples were examined by SDS-PAGE (2,5).

Bio Rad (BR) and Protein Gold (PG) assays for determination of proteins were compared. The ratio between BR and PG ranged from 1.8

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to 3.8. These values agree with those obtained from ammonium sulfate samples reported last month (4).

After screening the Stratagene cDNA library prepared from Poly(A+) RNA of tobacco root, plaques that strongly hybridize to root cDNA probe as compared to leaf cDNA probes were picked and plated individually for further screening. Stratagene's "RNA isolation kit" was used for preparations of total RNA from tobacco roots and leaves. The integrity of total RNA preparations was confirmed by electrophoretic analysis on formaldehyde agarose gel. Commercial mRNA purification kits were used to purify poly(A+) RNA from total RNA (6,7).

The ^{32}P-labeled first cDNA strands were synthesized using poly(A+) RNA from leaves and roots and were used to screen 75 preselected plaques with the hope of identifying cDNA sequences which are overly expressed in tobacco roots. Further work is needed to select DNA sequences which are overly expressed in roots as compared to leaves of tobacco (6,7).

B. **Plans**: Continue to harvest tobacco roots and prepare ammonium sulfate extracts. Fractionate PMT-containing samples by various methods to aid in purification. Continue to examine ServaNet IEF system for identification of PMT. Continue the isolation of cDNA sequences which are overly expressed in roots from tobacco root library.

D. **References**:

1. Lyle, J. Notebook No. 8856, p. 23,34.
2. Davies, S. Notebook No. 8907, p. 10.
3. Lyle, J. Notebook No. 8856, p. 57.
4. Yu, T. Notebook No. 8848, p. 92.
5. Nakatani, H. Notebook No. 8384, pp. 78-84.
6. Wahab, S. Notebook No. 8855, p. 61.
7. Malik, V. S. Notebook No. 8899, p. 11.

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PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: November 1989

I. **SUBJECT** Cigarette Delivery Modeling (D. Newman, B. Dwyer)

A. **Objective**: Develop computer programs to assist in designing cigarettes.

B. **Results**: A computer program has been written to evaluate the effects of bands of burn-control agents applied to cigarette papers. The program predicts the effects of the band positions and frequencies on puff count and tar delivery.

We are also working with Operations Services and Quality Assurance to develop criteria for incoming materials. In order to identify critical properties of such materials and quantify the effects of these properties on cigarette performance, we are using the delivery model to make predictions. Currently, we are evaluating the effects of wrapper permeability and citrate variations on tar and puff-count variabilities.

II. **SUBJECT** ART Processing Studies (D. Simpson, T. Howell)

A. **Objective**: Support the design and operation of the ART program.

B. **Results**: Two trips were made to Pemm Corp to visualize tobacco-bed fluidization in their apparatus. These results have assisted in our design of a gas distribution plate for the commercial process.

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PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: November 1989

I. **INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES** (Fournier, Kallianos, Paine, Podraza, Seeman)

A. **Objective**: To develop inorganic materials for novel applications for reduced sidestream, for burn-rate modification, and enhanced subjectives in cigarettes and for required properties in novel smoking materials.

B. **Results and Plans**: Work continues on sol-gel derived $MgCO_3$ particles: new samples were prepared, submitted for handsheet-making, the effects of drying/heating conditions on particle size were investigated (BET 236 m^2/g after heating to 450°), and dynamic light scattering was performed on the sol (average particle size of 118 nm). Zirconia gels coated (5%) on cigarette paper were not effective in the burn-rate test while the zirconia-acetylacetonate complex was effective.

Optimization of the magnesite ($MgCO_3$) preparation continues. To date, magnesite hydrothermally prepared from hydromagnesite ($Mg_5(CO_3)_4(OH)_2.5H_2O$) at 250°C for 14 hours has given the smallest particle size as determined by laser diffraction. Shorter periods of time result in the incomplete conversion. Approximately 70 g of magnesite prepared under the aforementioned conditions will be available this week for paper making.

Nesquehonite ($MgCO_3.3H_2O$), was prepared by adding magnesium chloride to a stirred and sonicated solution of potassium bicarbonate. Attempts to prepare nesquehonite without the use of sonication resulted in a mixture of $KHCO_3.MgCO_3.4H_2O$ and nesquehonite as confirmed by x-ray diffraction. This is a case where sonication aided the reaction. The retention of the nesquehonite in paper was low and papers were not made. It may find use as a coating.

A K/Fe phosphate was prepared hydrothermally. X-ray diffraction results to determine the actual phase are pending.

II. **REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS** (Paine, Secor, Seeman)

A. **Objective**: To develop techniques to remove selectively nicotine and other alkaloids from aqueous tobacco processing fluids.

B. **Results and Plans**: Development of data necessary to develop a pilot plant scale clay-mediated nicotine separation scheme is in full progress. Various clays are being examined, and the quantities of nicotine absorbed, sulfuric acid need to reextract the nicotine, and water washes are being determined. Clay

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lifetimes (i.e., recycle capabilities) are also being examined. In collaboration with A. Kumar, specialized equipment for back flow extractions have been designed and assembled. Various processes are being considered for potential commercialization, and interactions with outside vendors/consultants have been valuable.

III. **NICOTINE DESTRUCTION** (Podraza, Secor, Seeman)

A. **Objective**: To develop a commercial scale procedure for the chemical destruction of nicotine.

B. **Results and Plans**: Experimental studies on three nicotine destruction procedures have been very carefully examined, and work is nearly completed: hydrogen peroxide, sodium hypochlorite, and ozone. In conjunction with A. Frisch, U-^{14}C-nicotine has been used to follow mass balance in these three procedures, and in all cases, >98% carbon has been accounted for. The major and most of the minor products in the three methods have been identified, and some optimization in reaction conditions have been successfully performed. A full report is being written and patent disclosures are being prepared.

IV. **FLAVOR/ODOR CHEMISTRY** (Howe, Paine)

A. **Objective**: To prepare new substances for flavor/odor evaluation.

B. **Results and Plans**: In support of Y. Houminer's vanillin program, ca. 10 g of a vanillin release compound was prepared under a high priority-rush request by a novel route and transferred to Flavor Development.

V. **MISCELLANEOUS** (Secor)

Results: 2-(2-Hydroxyethyl)quinoline was prepared and sent to E. Bernstein as a model for the flavor release systems being developed for a variety of flavor objectives. Two 2,4-dinitrophenylhydrazones (of formaldehyde and acetaldehyde) were prepared for R. Comes as standards in the SS program.

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PROJECT NUMBER: 2501
PROJECT TITLE: SMOKE CHEMISTRY
PROJECT LEADER: R. A. COMES
PERIOD COVERED: November, 1989

I. SIDESTREAM SMOKE CHAMBER

A. **Objective**: Design, construct and operate an environmentally controlled chamber to measure selected components of sidestream smoke.

B. **Status**: All analyses of chamber generated samples (except for nitrosamines which are analyzed by BCR personnel) are now conducted by Project personnel utilizing Project instrumentation. Multiple chamber runs were carried out on C20 (European Monitor) and on Monitor-25 cigarettes. Sidestream data obtained included CO, CO_2, NO, ozone, particulate matter, ammonia, nicotine and nitrosamines. The data was reported in a memo issued 11/10/89.

Several Japanese brands were smoked during this reporting period (see Section II., below).

Cigarettes from the analytical/subjective study (X8D9XM, X8D9XO and X8D9XQ) were evaluated in the chamber. Results, including aldehydes, have been tabulated and a memo is in progress.

Methodology for data collection has been successfully developed for use with the instrument for determination of particle size distribution. Programs have been written for transfer to computer and for manipulation of the data utilizing mainframe capabilities. Samples analyzed for nicotine included those for the subjective/analytical study, Monitor-25's and a substantial number of C20 reference cigarettes. They were analyzed by both the XAD and Extrelut procedures. The C20 run data using both methods has been submitted to PED for statistical analysis to answer questions regarding the suitability of the sampling protocol, the acceptability of experimental variation and the feasibility of dropping the Extrelut analysis procedure. The data is currently being analyzed and a report will be issued. The Hewlett-Packard gas chromatograph with NPD and FID detectors and automatic injector has been installed and checked out in the chamber laboratory. It will shortly become the instrument of choice for nicotine analysis. A Hewlett-Packard HPLC has been ordered to facilitate aldehyde analyses. A request has been made and is currently being acted on for the synthesis of new aldehyde and ketone 2,4-DNPH standards by Project 2500 personnel. A new water deionizer system has been installed for use with the ammonia specific ion electrode procedure. Linearity checks using reagents prepared with water from the new system were acceptable. Plans have been formulated and pertinent information forwarded to the vendor to facilitate shipment of the transmissometer. In-house construction of the required stand to mount this instrument will commence shortly. The Beckman CO_2 monitor discussed in the previous report was attached to the mainstream outlet from the

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chamber smoking machine. Attempts at monitoring chamber CO_2 during smoke runs was successful in that a response was observed, but unsuccessful in that the read-out was extremely noisy. Other monitors for CO_2 and CO will be investigated. Mainstream TPM data can be collected using the large TPM pad supplied with the 30-port smoker.

C. **Plans**: Chamber runs will continue as required. A new monitor, IM13, is currently being evaluated using all chamber parameters. The transmissometer should be delivered early in December and will be installed and placed into routine operation. The third instrument for generating cigarette smoke particle information (total number of particles) will be investigated shortly.

II. SIDESTREAM SMOKE

A. **Objective**: Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream semivolatiles and gas phase; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.

B. **Status**: A series of Japanese cigarettes (Mild Seven Super Lights, Mild Seven Lights, Mild Seven, Lark Super Lights and Lark) was investigated on the prototype Coresta smoking machine and in the sidestream chamber to determine mainstream and sidestream smoke data. Three runs of each test model were carried out in the chamber to generate sidestream particulate data using the TEOM apparatus. Monitor-25 TEOM data was obtained on four replicates. Duplicate runs were conducted utilizing the Coresta smoking machine except in the case of Monitor-25 and Mild Seven where three runs each were carried out. Coresta data generated included static TPM, static burn times, dynamic TPM (mainstream and sidestream) and puff count. The information was compared with CI TPM and visibility data and was reported in a memo issued 11/7/89.

III. MISCELLANEOUS

1. An analysis was carried out on products derived from a nicotine destruction experiment. The results were reported in a memo.

2. Gc/ms and pyrolysis/gc/ms analyses were conducted on various samples as requested. Memos are being written in support on these analyses.

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PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: Y. Houminer
WRITTEN BY: R. Southwick
PERIOD COVERED: November, 1989

I. COMMERCIALIZATION OF GMC AND POLYMIC

A. **Objective**: To find chemical manufacturers who are capable of producing large quantities of GMC and polyMIC.

B. **Results**: GMC produced by John Paine from menthylchloroformate (MCF) in toluene has been found acceptable both analytically and subjectively. Accordingly, a production run of MCF from 1821 pounds of --menthol was carried out by PPG and the material shipped as a toluene solution to Lee Labs. The solution was found analytically acceptable; about 1610 pounds of MCF were produced.

A trial production of 250 pounds of GMC at Lee Labs was begun on November 12. An unanticipated operational failure rendered the entire batch unsuitable for use. Following careful review of all procedures, a second trial will begin during the week of November 26. This material will be available in the first week of December.

II. FLAVOR RELEASE TECHNOLOGY

A. **Objective**: To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

B. **Results**: In collaboration with Project 2500 (J. Paine and C. Howe), a large scale preparation of CR-2852 was accomplished. The material (15g) was submitted to Flavor Development for further evaluation as a vanillin sidestream flavoring agent. This large scale preparation was accomplished by two separate routes: one utilized a THP-protected vanillin and the other a benzyl-protected vanillin. Each was condensed with ethyl phenylacetate to yield the corresponding protected hydroxy-esters, which were subsequently de-blocked to give CR-2852. An attempt to carry out a direct large-scale synthesis by condensation of ethyl phenylacetate with unprotected vanilliin failed to give the desired product.

An alternate vanillin-release agent (CR-2887) is now available for evaluation by Flavor Development. This compound, a vanillin ester of CR-1719, upon pyrolysis at 300° cleanly yields vanillin and a tobacco-identical lactone. A patent disclosure discussing these results has been submitted.

The preparation of a vanillin ester of gluconic acid continues to provide synthetic challenges. Reaction of di-isopropylidene gluconic acid with vanillin under various conditions failed to

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give any of the desired ester. In the belief that the alpha-hydroxyl group may be interfering with esterification, blocking groups for this free hydroxyl are being investigated.

The analysis of mainstream, sidestream and butts from cigarettes (heavy paper, code X8D9DAU) made from paper coated with Aromatek 245 has been completed. Total accountability of mass was 65%. The amount of Aromatek 245 decomposing to deliver alpha-hexylcinnamaldehyde as well as the distribution of the compounds to MS and SS were nearly identical to that found in a previous study using Marlboro Lights. The sidestream contained 90% of the released aldehyde as well as 80% of the recovered (intact) Aromatek 245.

III. FLAVOR CHEMISTRY

A. **Objective**: To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.

B. **Results**: Data from the newly-established Trigeminal panel have been analyzed and the following conclusions drawn:

1. The data are adequately reproducible;

2. The descriptors being used can separate olfactory and trigeminal stimulants clearly;

3. The descriptors can separate Trigeminal stimulants such as nicotine, acetic acid and menthol;

A memo describing these results in greater detail is in preparation. Evaluation of other stimulants continues.

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PROJECT NUMBER: 2525
PROJECT TITLE: Paper and Tobacco Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: November, 1989

I. SIDESTREAM REDUCTION (S. Baldwin, S. Tafur, G. Bokelman, J. Kang, G. Newell, P. Suiter and B. Rogers)

A. **Objectives**: (1) Prepare and evaluate, for reduction in sidestream smoke, paper handsheets that contain additives and/or inorganic fillers with different physical and chemical properties and (2) evaluate machine-made cigarettes containing cigarette papers produced at the University of Maine or by Kimberly-Clark.

B. **Results**: An U.S. patent application was filed which, among other claims, covered the use of KH_2PO_4 on high basis weight cigarette papers.

Sherry Baldwin and Gordon Bokelman visited Dr. Helena Chum, an expert on cellulose and biomass pyrolysis, at the Solar Energy Research Institute (SERI) in Golden, Colorado. Prior to the visit two samples, representing high basis weight cigarette paper with and without KH_2PO_4 additive, had been submitted for preliminary analysis on the molecular beam mass spectrometer at SERI. The results indicated distinctive differences between the samples. This information may be useful in understanding how paper additives affect subjective characteristics, as well as reduce sidestream. The speed and ease with which real-time analysis of pyrolysis products can be achieved in the SERI laboratory is most impressive. A memo was prepared which gives more details on this trip and also includes a preliminary proposal for research to be conducted at SERI by Dr. Chum's group (1).

Three high basis weight papers made at the U. of Maine and sized with different levels of KH_2PO_4 were used to prepare handmade cigarettes for sidestream analyses. Optimal sidestream reduction was clearly a function of paper porosity. These models were designed to provide assistance for commercial development of a low sidestream paper by Kimberly-Clark.

The examination of a number of different chemical sizings on medium and high basis weight papers was continued. A soluble form of "ferric" pyrophosphate (probably mixed ferric and ferrous) has been shown to be quite effective for reducing sidestream at relatively low levels (~3%). Problems with this sizing include a slight yellow color which is imparted to the paper and the production of a reddish ash. Further examination of other iron salts (mostly ferrous) is planned as well as investigation of the potential for double-wrapping to hide the problems with "ferric" pyrophosphate. Reduction of sidestream was also shown with tetrapotassium pyrophosphate, but at higher levels (7-8%) than the "ferric" form. High levels of MAP and DAP (13-17%) produced no significant sidestream reduction and gave very long static burn times. A combination of KH_2citrate and KH_2PO_4 gave very good sidestream reduction with a faster static burn time than KH_2PO_4

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alone at a comparable K+ level. The addition of monobasic calcium phosphate with KH_2PO_4, to provide soluble Ca++ for potential cross-linking with polymeric metaphosphate formed upon heating, showed no advantage. In another study, potassium gluconate sizing was found to be effective in reducing sidestream.

Replacement of 5 to 20% of the flax fiber in paper with microcrystalline cellulose (Avicel) produced no effect on sidestream reduction. These papers burned faster, which may be a useful feature for off-setting chemical sizings that give long burn times. These papers also displayed good formation and had a unique "soft" feel.

A bilayer paper was examined in an attempt to improve the ash appearance of paper with hydromagnesite filler. A lightweight layer with 10% Multifex MM $CaCO_3$ was added to the hydromagnesite layer and handmade cigarettes were prepared with the $CaCO_3$ layer on both the inside and outside of the cigarettes. Ash appearance was better with the $CaCO_3$ layer on the outside, but the sidestream reduction was only moderate. Additional bilayer configurations need to be examined to determine the best combination of inorganic fillers and basis weights of the layers.

A mathematical model was developed using data obtained from a previous matrix study of high basis weight cigarette papers. Predicted values for sidestream reduction performance based on this regression model for three U. of Maine papers and one paper from Kimberly-Clark were in good agreement with experimental values. In order to extend the applicability of this model to machine-made cigarettes, data will be generated to develop a correlation between single port and eight-port visibility measurements for machine-made cigarettes.

Preliminary experiments have indicated that the addition of phosphoric acid to the size gives further improvements in sidestream reduction for cigarettes with high basis weight papers containing KH_2PO_4.

In another set of experiments, thermocouples were placed on the surface and also inserted within the paper wrapper of low sidestream cigarettes. During static burn the maximum temperature achieved by most papers was between 500° and 550° C. In general, it was found that the shorter the static burn time, the higher the paper temperature and that higher basis weight papers gave higher temperatures.

A study of the surface area of paper char was initiated (2). Preliminary experiments involved developing a method for analysis and examining the effects of various chemical additives on paper. The results to date suggest that increases in surface area of paper char alone cannot account for sidestream reduction.

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For the month, 49 cigarette models were handmade and sidestream analyses were obtained on 54 models. These models included papers made with magnesite ("true" $MgCO_3$) and papers made by dispersing the $CaCO_3$ filler in different modes.

C. **Plans**: Numerous cigarette models will be prepared having medium (~45 g/m^2) basis weight, low permeability wrappers and examined for sidestream reduction and subjective characteristics. In addition, four new high basis weight cigarette papers prepared at our request by Kimberly-Clark will be evaluated. Paper handsheets containing $MgCO_3$ that was prepared by Andy Kallianos via sol-gel techniques also will be evaluated. Studies will be continued on the BET surface areas of paper chars. Finally, the paper database will continue to be modified and updated.

D. **References**:

1. Baldwin, S. D. and G. H. Bokelman, memo to R. N. Ferguson, "Pyrolysis/Combustion Experiments on High Basis Weight Papers at the Solar Energy Research Institute," November 13, 1989.

2. Tafur, S. and J. Kang, memo to R. N. Ferguson, "BET Surface Areas of Paper Chars: Method Development and Initial Results," November 17, 1989.

II. GREENHOUSE STUDIES & SUPPORT ACTIVITIES (R. Bass, G. West & G. Newell)

A. **Objectives**: Maintain the R&D greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs and provide requested assistance for special projects.

B. **Results**: 371 flue-cured tobacco breeding line samples have been received and submitted for alkaloid and reducing sugar analyses.

Tobacco processing and cigarette fabrication for the seven samples of the 1989 Regional Farm Test on Flue-Cured Tobacco have been completed. As scheduled, the cigarette samples have been distributed to various companies for subjective evaluation.

The hydroponic Burley 21 plants of Group 21 for Project 1904 were harvested. The group 22 plants will be seeded soon.

Fifty tobacco-less cigarette tubes were prepared for Robert Jenkins to be used in neutron activation studies of sidestream smoke.

C. **Plans**: Maintain plant production schedules. Inspect and monitor plant and environmental conditions on a daily basis.

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PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
PERIOD COVERED: November, 1989

I. PROJECT ART

A. Objective: Measure mainstream deliveries of gaseous ammonia by TDL and total ammonia by ion chromatography, and use the ratio of NH_3 to NH_4+ to calculate a value proportional to the basicity of ART cigarette smoke.

B. Results: The ammonia deliveries of ART cigarettes, when measured by both the TDL and the ion chromatography procedures, can be divided into gaseous ammonia (NH_3) and ammonium ion (NH_4+). A relative value of basicity of the smoke can be calculated using the ratio of NH_4+/NH_3 and the pK_a of ammonia. The table below shows the results of these calculations for a monitor #25, a production Merit ,and a production ART from the manufacturing Center.

Sample	Gaseous Ammonia (µg/cigt.)	Ammonium Ion (µg/cigt.)	Basicity (~pH)
MON #25	2.1	26.9	8.13
Merit 85	1.5	17.5	8.17
ART(X9FP)	4.0	26.0	8.43

C. Conclusions: The basicity of the smoke of ART production model as expressed in pH units was higher than both Merit and Monitor #25 Cigarettes. This result agrees with previous studies which have shown that ART cigarettes have higher deliveries of basic components such as pyridines and pyrazines in MS smoke condensate.

D. Plans: No further work in this area of investigation is planned. The project will however support requests for total ammonia in MS smoke.

E. References:

Parrish, M., Notebook #8858, p. 40.

II. MAINSTREAM AND SIDESTREAM SMOKE STUDIES

A. Objective: To develop a method for multicomponent quantitative analysis of the gaseous components of mainstream smoke using FT-IR spectroscopy.

B. Results: The deliveries of acetaldehyde, HCN, NO, and CO for a monitor #25 were measured using a newly developed method for gaseous components in MS smoke based on quantitation by FTIR spectroscopy. The measurements were made at a relatively low

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spectral resolution of two wavenumbers to mimic the ability of low cost FTIR spectrometers to use the method. Absorption peaks for each gas were selected that varied linearly with concentration within the range found in cigarette smoke. A dilution factor of approximately 7 brought all four gases within the linear range. Sets of 5 monitor M25 cigarettes were smoked on two separate days. The deliveries of acetaldehyde, HCN, NO, and CO as measured by this method were 850±20 µg/cigt, 210±10 µg/cigt., 300±10 µg/cigt., and 15.5±5 mg/cigt. respectively. Except for the delivery of acetaldehyde these values are in excellent agreement with the gas phase deliveries as measured by reference methods. The concentration of acetaldehyde is approximately 15% high compared to values measured by dinitro-phenyhydrazone derivatization and HPLC analysis. This is due to interferences from other smoke aldehydes and ketones.

C. **Conclusions**: The FTIR analysis method for HCN, NO, and CO has been shown to be a suitable method of quantitation of these components in MS gas phase. However, further studies need to address the selection of spectral bands to more accurately quantify acetaldehyde.

D. **Plans**: To purchase a low cost FTIR and interface it with a five port smoking machine such as is used in CI. Possible improvements in the data analysis algorithms will be investigated. Programs will be written to automate the analysis procedure. The feasibility study results will be presented to Jane Lewis.

E. **References**:

Koller, K., PM Notebook 8870.

A. **Objective**: To develop methodology for studying SS odor and irritancy

B. **Results**: A sidestream collection chamber, designed by B. Jenkins, was positioned in line with a cambridge filter pad. This arrangement first entrained the whole smoke, then the pad removed the particulate phase of the smoke isolating the vapor phase components for further study. A gas phase fractionation system was interfaced to this collection system to collect the gas phase components for GC analysis. The fractionation system was designed based on the breakthrough phenomenon of polymeric adsorbents and cryogenic trapping. Glass tubes containing adsorbents of differing surface areas, Carbotrap C (12 m^2/g) and Carbosieve (550 m^2/g), were positioned after the Cambridge pad. At a given flow rate, compounds with higher volatility are subjected to less retention and therefore, exit from the adsorbent tube. By varying the amount of adsorbent, fractions containing compounds with different volatility were isolated. The gaseous stream exiting the system can be sampled via a heated gas sampling valve at different time intervals and injected into a gas chromatograph for

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analysis via cryogenic focusing. At the exit port of the sampling valve, various fractions of the SS gas phase were evaluated for their odor properties. Those compounds retained by the adsorbents or cold traps were also evaluated.

C. **Conclusions**: The fractionation methods using either adsorbents or cold traps offer the flexibility to combine fractions to study the synergistic effects. With charcoal filters, the fractions which contained highly volatile compounds gave ammonia-like, fishy and pungent odor. Fractions with higher complexity, isolated by Carbotrap C , were very harsh and irritating in addition to the ammonia-like odor. Compounds retained by the Carbotrap had odors which were highly smoky and stale, similar to ashtray aroma.

D. **Plans**: Continue the fractionation experiments and establish the relationship of odor/irritancy versus volatility and functionality.

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PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: November, 1989

I. PROJECT ART

A. **Objective**: Provide analytical support to project ART.

B. **Results**:

Filler and on-line samples from the 1-L extraction system were analyzed for nicotine. Results from these analyses were reported to Tony Howell.

A method for the determination of nicotine in aqueous solutions of monopotassium citrate was developed for use at BHPP. The method gives quantitative recovery of nicotine and uses the same standards and extracting solution of Method E-86A.

C. **Plans**: Continue to support Project ART as needed.

II. ORGANOCHLORINE PESTICIDES METHOD

A. **Objective**: To explore alternative methods for the determination of organochlorine pesticides in off-shore tobaccos.

B. **Results**: Using 1.0 g of tobacco and an extraction time of 2 hours with 5% dichloromethane in hexane gave recoveries of at least 80% for all pesticides. The use of the smaller sample size of 1.0 g vs. 5.0 g results in a cleaner baseline with fewer negative peaks.

C. **Plans**: Investigate the possibility of using solid phase extraction for sample cleanup.

III. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

B. **Results**: Analyses and investigations by project personnel during the month of November included:

Several customer complaint samples were analyzed.

Quantitative and qualitative analyses of various packaging materials were performed for Rick Dunaway and Packaging Technical Services.

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Various flavors and flavor release compounds (Aromateks and related compounds) were quantitated on cigarette paper for Flavor Development.

Numerous tobacco samples were analyzed for methoprene. One sample from Warehouse 28 was analyzed for R- and S- methoprene. The separation was performed on a normal phase, chiral column after collection of the methoprene from several HPLC runs. Integration of the two isomers showed that equal amounts of R- and S-methoprene were present. Results were reported to L. Ryan and/or D. Faustini.

A rapid, HPLC method for the quantitative determination of methoprene which was developed last month was investigated further. Recoveries of methoprene from tobacco fortified with levels from 0.25 to 20 ppm ranged from 96.3% to 99.4%. The standard deviation for 10 samples fortified at the 3.4 ppm level was 0.1 ppm.

Samples of Spanish Asta and Venezuela feedstock were analyzed for acetic, propionic, and butyric acids. Results were reported to Grant Gellatly.

An off odor in an adhesive sample submitted by Pat Grantham was determined to be largely due to the presence of 3-hydroxy-2-butanone.

Minor alkaloids were determined in several tobacco samples.

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PROJECT NUMBER: 6902
PROJECT TITLE: Biochemical Special Investigations
PROJECT LEADER: B. D. Davies
PERIOD COVERED: November, 1989

I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

A. **Objective**: To obtain a monoclonal antibody (MCA) against nicotine (NIC-MCA).

B. **Results**: It was determined that clone BE2 antibodies are of the IgM class. This completes BE2s characterization, a memo describing the complete characterization will be issued.

Screening was begun on media from several new clones. Three of ten clones possessed the required titer and levels of antigen specificity. One of these three, HA4, was further tested in the nicotine inhibition assay. Its 50% inhibition constant (IC50) was determined to be 3.98E-6 M.

An additional batch of succinylated hydroxyethylnicotine-human serum albumin (SHN-HSA) was synthesized and characterized.

C. **Plans**: Continue screening and characterizing positive clones. Extract tobacco with water at pH ~7 and 5% acetic acid, compare the efficiency of extraction by submitting both samples to ARD for nicotine analysis. Test the water extract in an ELISA. Send the SHN-HSA to Hazleton Laboratories for the production of polyclonal antibodies.

D. **Reference**:

Crockett, E. Notebook No. 8863, p. 111.

II. ADDITIONAL APPROACHES TOWARD PUTRESCINE METHYLTRANSFERASE (PMT) ISOLATION

A. **Objective**: Provide additional experimental approaches to assist in the effort to isolate PMT.

B. **Results**: Samples of photolyzed and nonphotolyzed PMT were fractionated on Phastsystem IEF gels. In this preliminary study, the photolyzed sample appeared smeared and only faint banding patterns were visible. This was in contrast to the untreated sample which (like the control) gave distinct banding patterns.

C. **Plans**: Conduct additional studies to determine the actual effects of photolysis of PMT material on its migration characteristics in IEF gels .

D. **Reference**:

Crockett, E. Notebook No. 8863, p. 111.

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PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: G. J. Patskan
WRITTEN BY: W. R. McCoy
PERIOD COVERED: November, 1989

I. GLUTATHIONE DEPLETION ASSAY

A. Objective: To evaluate the role of arachidonic acid metabolism in the reduction of glutathione by 2R1 CSC in V79 cells.

B. Results: Treatment with indomethacin (1 to 100 μM), an inhibitor of cyclooxygenase activity, showed variable effects on the reduction of glutathione by 2R1 CSC. Two previous experiments showed indomethacin to prevent glutathione reduction by CSC by approximately 40% and 70%. Most recent results showed indomethacin to increase glutathione reduction by CSC by up to two-fold. Treatment with nordihydroguaiaretic acid (NDGA; 0.1 to 10 μM), an inhibitor of lipoxygenase activity, prevented glutathione reduction by CSC by approximately 35%. A previous experiment had shown NDGA to prevent glutathione reduction by CSC by approximately 50%. No dose response was seen for indomethacin or NDGA. Indomethacin or NDGA alone did not alter cellular glutathione levels.

C. Plans: The testing of other inhibitors is under consideration.

D. Reference:

McCoy, W. R. Notebook No. 8739, pp. 185-187.

II. JB6 MOUSE EPIDERMAL CELL TRANSFORMATION ASSAY

A. Objective: To implement the JB6 mouse epidermal cell transformation assay in our laboratory.

B. Results: A third soft agar assay testing TPA was set up, and all three TPA tests were counted for colony formation. Two plate growth toxicity experiments using mezerein and two using phorbol diacetate were completed. Results correlated with dose levels reported in the literature for soft agar assays. One benzoyl peroxide soft agar experiment was completed. There appeared to be some problems with that particular test, but it was not possible to determine the precise source(s) of the difficulty from the experimental data. Two mezerein soft agar assays were begun.

C. Plans: Statistical analysis of the TPA results will be completed by J. Tindall. A second benzoyl peroxide soft agar assay will be set up in a manner designed to determine the source(s) of problems in the original experiment. A third mezerein soft agar assay will be started. An automatic colony counter will be evaluated for possible use in facilitating this assay.

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D. **Reference**:

Nixon, G. M. Notebook No. 8711, p. 149.

III. ACQUISITION AND MAINTENANCE OF CELL LINES

A. **Objective**: To acquire and maintain a variety of cell lines for use in biochemical assays.

B. **Results**: New frozen 3T3 cells from ATCC are doing well. Results from Microbiological Associates indicated that mycoplasma was found in our 3PC cell line, while no mycoplasma was found in all other cell lines tested. Another batch of the 3PC cell line and a sample of the MT 1/2 cell line were sent to ATCC for mycoplasma testing. The generation time of each JB6 cell clone has decreased. The JB6 cell clones are being monitored closely in conjunction with the transformation assay.

C. **Plans**: Continue to monitor each cell line.

D. **References**:

Burruss, T. J. Notebook No. 8896, p. 14.
Vaughan, B. G. Notebook No. 8828, p. 85.

IV. EGF BINDING ASSAY

A. **Objective**: To examine the role of phospholipid metabolism in the inhibition of EGF binding caused by CSC.

B. **Results**: A second experiment with ETYA, an inhibitor of both lipoxygenase and cyclooxygenase activities, was conducted this month. As with the first experiment, only a minimal effect of ETYA on EGF binding was observed following treatment with ETYA alone, and there were no significant effects of ETYA on the response of 2R1 CSC.

C. **Plans**: To examine the effects of PKC down regulation and the role it plays with CSC on EGF binding.

D. **Reference**:

Stagg, D. L. Notebook No. 8883, p. 66.

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PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: November, 1989

I. UNEXTRACTED NICOTINE STUDIES (with W. Hempfling)

A. Objective: To determine whether there is a correlation between unextracted nicotine (UN) in filler and the delivery of NNK in the corresponding MS smoke.

B. Results: Supercritical CO_2 extracted DBC-Bright, DBC-Burley, and MT-Oriental fillers were obtained from Physical Research Division, and values for TARN,[1] EN,[2] Nic-X[3] and Nic-Y[4] were determined using previously described methods. TARN for all the fillers was in the range 1200-1900 ppm, and EN varied from 40% of TARN (Or) to 61% of TARN (Br). Digestion of the hot water extracts gave Nic-X values of 190 ppm (Or) to 389 ppm (Br). Nic-Y values were then calculated by subtracting the total of EN + Nic-X from the TARN values. When the marcs from the water extractions were treated with hot alkali, direct measurements of Nic-Y were obtained. Unexpectedly, the direct Nic-Y values were only 42%, 35%, and 14% of the calculated Nic-Y values, respectively.

A possible explanation for these discrepancies is suggested by the data obtained for nornicotine (NN) on the same samples. TARNN[5] values for the three fillers ranged from 1214 ppm (Or) to 3233 ppm (Bu). ENN[6] varied from 62% of TARNN (Or) to 100% of TARNN (Bu). Amounts of NN-X[7] that were found after digestion of the hot water extracts in hot alkali were not statistically significant. All of the NN was extracted from burley with hot water, but 355 ppm of NN was still released from the hot water burley marc by hot alkali digestion. The sum of Nic-Y and NN-Y[8] nearly equaled the amount of Nic-Y expected (624 ppm). However, no NN-Y was found in the oriental marc, although ~400 ppm was expected.

C. Plans: Repeat the extractions of SCFE fillers and submit for analysis to confirm the results obtained above. Compare nicotine content in Bu-21 green samples as determined by our method with those found by pyrolysis (ARD).

D. References:

Lambert, E. A. Notebook No. 8852, p. 59.
Keene, C. K. Notebook No. 8754, p. 190.

E. Footnote:

[1]TARN = total alkali-releasable nicotine. [2]EN = nicotine extractable by hot water. [3]Nic-X = hot-water-soluble alkali-releasable nicotine. [4]Nic-Y = hot-water-insoluble alkali-releasable nicotine. [5]TARNN = total alkali-releasable nornicotine. [6]ENN = nornicotine extractable by hot water. [7]NN-X = hot-water-soluble

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alkali-releasable nornicotine. [8]NN-Y = hot-water-insoluble alkali-releasable nornicotine.

II. ORIENTAL INHIBITOR STUDIES

A. **Objective**: To determine whether the causative agent(s) responsible for the reduced levels of MS TSNA observed for Or tobacco can be removed with organic solvents or supercritical CO_2 and applied to other fillers as a means of decreasing the MS TSNA levels delivered by these fillers.

B. **Results**: The SCF CO_2 extract of blended Or filler, prepared at the facilities of Supercritical Processing, Inc., has been examined by TLC and an initial fractionation into three polarity categories has been performed. A major component was shown to be solanesol, which afforded no reduction in MS TSNA in earlier tests. Chemical classification of the subfractions is in progress, and additional material is being generated for further studies.

C. **Plans**: The fractions will be tested for inhibition of TSNA pyrosynthesis.

D. **Reference**:

Haut, S. A. Notebook No. 8891, p. 11.

III. TSNA REMOVAL STUDIES

A. **Objective**: To prepare a blend of fillers reduced in endogenous TSNA and minor alkaloid TSNA precursors, which can be used to make a laboratory cigarette with reduced MS TSNA delivery relative to that of a 1987 full-flavored, blended cigarette.

B. **Results**: DBC Bright and DBC Burley fillers were previously extracted with 95% ethanol using the cation exchange process to remove alkaloids and TSNA. Attempts were made this month to reapply the concentrated solids, minus the alkaloids and TSNA, onto the respective fillers in order to prepare the alkaloid and TSNA-depleted fillers as blend components. The initial attempt to spray the extract concentrates resulted in only a 30% recovery of solids. The second attempt gave better results, but the recovery of solids was still only ~75%.

C. **Plans**: Analyze the above fillers for alkaloids and TSNA, blend the burley and bright fillers with oriental, and smoke for MS TSNA.

D. **Reference**:

Tickle, M. H. Notebook No. 8716, p. 200.

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IV. TSNA PRECURSORS

A. **Objective**: To determine the precursors of MS TSNA.

B. **Results**: Fresh samples of Br and Bu RLs were obtained from R. Izac (Project 6912) for the purpose of initiating an RL aging study. Zero-time filler TSNA data were obtained. Samples that have been aged five weeks at 45°C have also been analyzed for filler TSNA, along with corresponding sampled stored at -30°C. The data obtained to date are inconclusive.

C. **Plans**: Further data on the RL aging study will be obtained. Plans are being made for a study of possible ways to remove UN from filler.

D. **Reference**:

Haut, S. A. Notebook No. 8891, p. 11.

V. MISCELLANEOUS AND SUPPORT STUDIES

A. **Objective**: To conduct studies of the TSNA content of filler and/or MS smoke or carry out other activities as necessary to support other PM programs.

B. **Results**: SAVA smokings of test cigarettes were conducted to determine whether various cigarette papers had any effect on either MS or SS nitrosamine levels. No differences due to cigarette paper were found.

C. **Reference**:

Morgan, W. R. Notebook No. 8905, pp. 1-2.

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PROJECT NUMBER: 6912
PROJECT TITLE: Tobacco/Smoke Relationships
PROJECT LEADER: S. B. Hassam
WRITTEN BY: S. Drew
PERIOD COVERED: November 1989

I. CROSS SOLUBLES/BASE WEB (CHEMISTRY)

A. **Objective**: Evaluate CSBW materials.

B. **Results**: Production of the base web, CEL and RL from bright, burley and oriental tobaccos has been completed.

A study involving the following precipitating agents: silver nitrate, silicotungstic acid and flavianic acid has been initiated. To remove nitrogenous components varying amounts of the three materials were added to a BuS1 sample produced from an earlier batch of Burley CEL. The decanted liquor from the resulting mixture is being sprayed on BrBW.

The BuS1 fraction is high in ninhydrin reactive material (NRM). Oxidation of NRM to remove nitrogenous compounds might result in lower biological activity. Attempts to use ozone to oxidize BuS1 were unsuccessful.

Electrodialysis of Bu CEL showed no selectivity for nitrates. Ninety percent of all the cations and anions were removed. A sample of the dialyzed Bu CEL was freeze dried to produce 24.8 grams of solids. The control sample was applied to 25.0 grams of BrBW as is.

Preparation of CSC from 68 glucose, fructose, sucrose and amino acid addition samples was completed.

C. **Plans**: Spray BrBW with samples from various treatments to produce model fillers. Request handmade cigarettes and replicate IT smoking. Prepare IT CSC samples and submit for S/M assay testing.

D. **References**:

1. Izac, R. Notebook No. 8874, pp. 15-25.

2. Drew, S. Notebook No. 8800, pp. 63-68.

3. Hellams, R. Notebook No. 8613, p. 193.

II. MAINSTREAM--SIDESTREAM--BUTT ANALYSIS

A. **Objective**: Evaluate mainstream, sidestream, and butts from cigarettes coated with Aromatek 245 for α-cinnamaldehyde delivery.

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B. **Results**: An analysis of mainstream, sidestream and the butts from cigarettes prepared with heavy paper and coated with Aromatek 245 was completed. The amount of Aromatek 245 decomposing to deliver α-cinnamaldehyde as well as the distribution of the compounds to the MS and SS was similar to that seen in a previous study (1).

C. **References**:

1. Izac, R., Core, M., Houminer, Y. Memo to Ferguson, R.N. August 3, 1989. Analysis of mainstream, sidestream, and butts from cigarettes coated with Aromatek 245.

2. Izac, R. Notebook 8874, pp. 15-25.

III. SUPPORT FUNCTION: SAMPLE PREPARATION

A. **Objective**: To prepare chemical fractions and/or condensates as needed for biological and chemical analysis.

B. **Results**: Several TLC plates were analyzed using a radio-TLC scanner. The TLC plates contained the reaction products of ^{14}C-(U)-nicotine with either ozone, hydrogen peroxide or sodium hypochlorite. The starting material, ^{14}C-(U)-nicotine, was analyzed and found to be 98% pure.

Twenty-two sets of handmade cigarettes were prepared for various projects within the BCR division. Cigarettes from 69 different samples were smoked on a 30-port Borgwaldt smoking machine for Project 6912. Three smokings were completed for Project 6906. Six tobacco samples were shredded for Project 6912.

C. **References**:

1. Izac, R. Notebook No. 8874, pp. 15-25.

2. McGee, N. Personnel Communication to S. Drew.

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PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Richard A. Thesing
PERIOD COVERED: November, 1989

I. MARKET ACTIVITY

A. **Objective**: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. **Results - Domestic**:

Lorillard is test marketing Harley Davidson 85 and Harley Davidson Lights 85 cigarettes in Arkansas. This is the first branded generic product for Lorillard. These cigarettes are packaged in a black pack with a gold eagle embossed on the pack. Harley Davidson delivers 11 mg tar and 0.8 mg nicotine; Harley Davidson Lights delivery 8 mg tar and 0.6 mg nicotine. Lorillard test marketed a Harley Davidson as a full price brand in September, 1987 in Terre Haute, Indiana.

The American Tobacco Company is distributing American Lights 100 Menthol cigarettes in Virginia. This branded generic product delivers 11mg tar, 0.9mg nicotine and 0.6mg smoke menthol. American currently has five versions of the American product on the market. American Lights 85 and 100; American Lights Menthol 100; and American Filters 85 and 100.

Results - International

Beside King Size cigarettes were introduced in Japan in October by Japan Tobacco, Inc. This product delivers 9 mg tar and 0.9 mg nicotine and has a dual carbon in CA/CA filter. Freon® was detected in the blend of this brand, indicating the presence of Freon® expanded tobacco.

Japan Tobacco, Inc. introduced **Peace International 95** cigarettes to the Japanese market in October. These cigarettes deliver 13 mg tar and 1.2 mg nicotine. Peace International has a single CA filter and is packaged in a princess box. The blend contains no reconstituted tobacco. Freon® was detected in the blend of this product, indicating the presence of Freon® expanded tobacco. JTI also markets Peace (NF), Peace King Size, and Peace Lights King Size cigarettes in Japan.

Peter Stuyvesant King Size cigarettes were introduced to the Japanese market in October by the American Cigarette Company (Overseas), Ltd., a subsidiary of Rothmans. This brand has a dual carbon in CA/CA filter and delivers 10 mg tar and 0.8 mg nicotine.

The Kumming Cigarette Factory of China introduced **Shiwn King Size** cigarettes to the Japanese market in October. This product delivers 13 mg tar and 0.8 mg nicotine. These cigarettes have a

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dual carbon in paper/CA filter. The blend contains 21% expanded stem, and no reconstituted or expanded tobacco.

San Chl King Size cigarettes were introduced to the Japanese market in October by the Yuxi Cigarette Factory of China. This brand has a single CA filter and delivers 20 mg tar and 1.0 mg nicotine. The blend of San Chl contains no reconstituted tobacco, expanded stem or expanded tobacco.

II. ANETHOLE ANALYSIS

The analysis for trace level anethole in filler and aftercut has been transferred from Flavor Development to CTSD. Equipment and supplies for the procedure have been ordered. CTSD personnel have been trained to run the analysis. Incoming samples will be analyzed in the Flavor Development area by CTSD personnel.

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